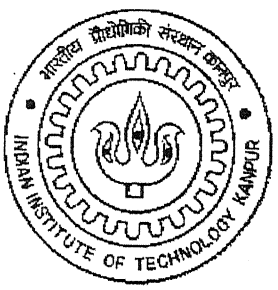
EXPERIMENTAL STUDY ON THE TEMPERATURE STEP RESPONSE OF MMA POLYMERIZATION IN A RHEOMETER-REACTOR ASSEMBLY

BY

SWARNENDU ROY



DEPARTMENT OF CHEMICAL ENGINEERING

INDIAN INSTITUTE OF TECHNOLOGY KANPUR

AUGUST, 2002

EXPERIMENTAL STUDY ON THE TEMPERATURE STEP RESPONSE OF MMA POLYMERIZATION IN A RHEOMETER-REACTOR ASSEMBLY

A Thesis Submitted
in Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY

BY
SWARNENDU ROY

to the

DEPARTMENT OF CHEMICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY KANPUR
AUGUST, 2002

DEDICATED TO

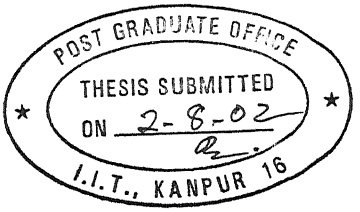
MY LIFE

MY PARENTS

AND

MY LOVE

KUMKUM



CERTIFICATE

It is certified that the work contained in the thesis entitled ***"EXPERIMENTAL STUDY ON THE TEMPERTURE STEP RESPONSE OF MMA POLYMERIZATION IN A RHEOMETER-REACTOR ASSEMBLY"*** by **Swarnendu Roy**, has been carried out under our supervisions and that this work has not been submitted elsewhere for a degree.

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NOMENCLATURE

M_w	weight average molecular weight (kg mol^{-1})
$x_m(t)$	monomer conversion (molar) at time t
k_d, k_p, k_t	rate constants for initiation, propagation and termination in presence of the gel and glass effects (s^{-1} or m^3 mol^{-1} s^{-1})
k_{tc}, k_{td}	rate constants for termination by combination or by disproportionation in the presence of presence of the gel effect (m^3 mol^{-1} s^{-1})
	initiator efficiency at time t
T_g	glass transition temperature (K)
$T(t)$	temperature of the reaction mixture at time t (K or ^{0} C)
$\eta(t)$	viscosity of the reaction mass (Pa s)

ABSTRACT

A rheometer-reactor consisting of a stainless steel cup and bob assembly (Haake, SV 2) has been used for the on-line measurements of viscosity during the bulk free radical polymerization of methyl methacrylate (MMA). Two different initiator [2,2'-azoisobutyronitrile (AIBN)] loadings (I_0=15.48 and 25.80 mol/m^3) are used, and data obtained under both isothermal and non-isothermal conditions. The viscosity of the reaction mass is obtained as a function of time during until reasonably high values of viscosity of about 41,000 Pa s are attained (falling well into the gel-effect region). The temperature requirements of isothermal as well as non-isothermal conditions are programmed and achieved by circulating water (from a circulating bath) through the chamber in a specially designed cup of the viscometer. Removal of the (exothermic) heat of reaction has been achieved by a similar procedure of circulating water at ambient temperature through this chamber, when required. The rheometer-reactor assembly provides signals to a measuring system interfaced with a PC over an RS-232 serial line through the rheocontroller. A Pt100 sensor placed in the reaction mass measures the temperature history.

CHAPTER 1

INTRODUCTION

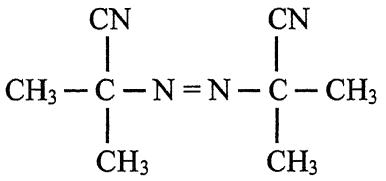
The molecular weight distribution (MWD) is the most crucial aspect in determining the physical properties of polymers, specially the commodity polymers. It is desirable to have high weight average molecular weight (M_w) polymers, having narrow MWD for wide range of applications. It is well established by Martin et. al.(1) and Nunes et. al.(2) that narrowing the MWD improves the thermal properties, stress-strain relationship, impact resistance, hardness and strength of the polymers. To produce polymers of desired quality it is required to measure the 'state' of the reaction mass, e.g. monomer conversion (x_m) and molecular weight (M_w)etc, continuously during polymerization reaction in the industrial reactor. Most industrial reactors for the manufacture of commodity polymers operate under non-isothermal, semibatch reactor conditions. The operating conditions are usually decided semi-empirically and possibly have some optimality criteria as their basis. Often, these criteria are intuition-based. There is a scope for improvement of the operating conditions in most cases, and the vast knowledge-base now available in polymer reaction engineering can be exploited to a considerable advantage. This study is a step forward along this direction for bulk free radical polymerizations, using poly(methyl methacrylate), PMMA, as a representative sample.

The basic kinetic scheme characterizing several important free radical polymerizations [e.g., PMMA, polystyrene (PS), poly(vinyl chloride) (PVA), etc.] is shown in Table 1.1. The three most important rate constants, k_d, k_p, and k_t ($\cong k_{td}$ for PMMA, since $k_{tc} \cong 0$) are associated with diffusional limitations at high monomer conversions.

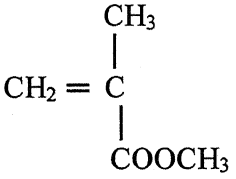
Table 1.1 Kinetic Scheme for Free Radical Polymerizations (Bulk and Solution)

Initiation	$I \xrightarrow{k_d} 2R$
Propagation	$R + M \xrightarrow{k_i} P_1$
Propagation	$P_n + M \xrightarrow{k_p} P_{n+1}$
Termination by combination	$P_n + P_m \xrightarrow{k_{tc}} D_{n+m}$
Termination by disproportionation	$P_n + P_m \xrightarrow{k_{td}} D_n + D_m$
Chain transfer to monomer	$P_n + M \xrightarrow{k_f} P_1 + D_n$
Chain transfer to monomer	$P_n + S \xrightarrow{k_s} S + D_n$
via solvent	$S + M \xrightarrow{fast} S + P_1$
	or
	$P_n + M \xrightarrow{k_s} D_n + P_1$

I = Azobisisobutyronitrile (Initiator)



M = Methylmethacrylate (Monomer)



The diffusional effects on these three reactions are referred to as the cage, glass, and gel effects, respectively. The cage effect causes a decrease in the initiator efficiency, f, at high monomer conversions. Several workers (3, 4, 5, 6, 7) assumed f to be constant with increasing monomer conversion. Russel et al. (8) and Achilias and Kiparissides (9,10) modeled the decrease of f with increasing monomer conversion. At higher monomer conversions, the propagation reaction also becomes diffusion controlled (glass effect). This effect is manifested when the glass transition temperature, T_g, of the polymerizing system becomes higher than the polymerization temperature, T, thus freezing segmental motion and stopping the reaction short of complete monomer conversion. The reaction mixture becomes glassy in nature beyond this point. The gel effect, first studied by Norrish and Smith (11) and Trommsdorff et al. (12), represents the effect of decreasing diffusivity of macro-radicals (due to increasing viscosity of the reaction mass) on the termination rate constant, k_{td} (and/ or k_{tc}), and is exhibited as a sharp increase in the monomer conversion, x_m, as well as the weight average molecular weight, M_w, with time, t.

Several workers have attempted to model the gel, glass and cage effects theoretically. Ross and Laurence (13) used concepts of free volume to express the apparent rate constants in terms of simple equations involving monomer conversion and temperature and curve-fitted the parameters of their model. Chiu et al. (4) presented a model (CCS model) which had several advantages over earlier models. Achilias and Kiparissides (9,10) proposed an improved model based on the generalized free volume theory of Vrentas and Duda (14,15) and the theory of excess chain-end mobility (6). Neither of these sets of theories could be applied to semibatch reactors. Ray et al. (16) and Seth and Gupta (17) developed a new model valid for both batch as well as semibatch reactors operating under more general non-isothermal conditions. The model predictions were confirmed experimentally (18,19) on bulk

polymerization of methyl methacrylate (MMA) for non-isothermal operation as well as with intermediate addition of initiator. This model can be used for on-line optimizing control of semibatch, non-isothermal reactors.

The control of polymerization reactors in the presence of severe diffusional limitations (as in bulk polymerizations) is expected to pose a formidable challenge, since these reactors are associated with an extremely fast increase in the monomer conversion with time after the onset of the gel effect. Such rapid changes in $x_m(t)$ leave almost no choice to the control engineer except to use models having excellent predictive capabilities. Model-based control is, thus, essential for the proper control of such reactors (20), since often one has to take control action (like changing the temperature) much before diffusional effects manifest themselves. The control of free radical bulk polymerization reactors poses yet another problem, namely, lack of suitable measuring instruments which can be used on-line. Use of gel permeation chromatography/ HPLC to obtain the molecular weight distribution, as well as the use of available densitometers for estimating monomer conversion is limited to solution polymerizations, and these cannot be used easily for bulk polymerizations. An easier way out is to develop a model, usually semi-empirical, which can predict the desired polymer qualities using easily measurable secondary variables (21). Such models serve the same purpose as hardware sensors and are called "software" sensors. Crowley and Choi (22) used a low-frequency dielectric sensor for in-line monitoring of monomer conversion during solution polymerization of MMA in laboratory-scale batch reactor. Buruaga et al. (23) used on-line calorimetry to estimate monomer conversion and copolymer composition in emulsion polymerization systems. The key step in inferential estimation of the state of the system is to establish a relationship between the difficult-to-measure quantities and more easily measured variables. Jo and Bankoff (24) developed an on-line viscometer some years ago, to measure

the variation of viscosity in a CSTR in which solution polymerization was carried out. Seth and Gupta (17) explored the use of experimental values of the viscosity, $\eta(t)$, of the reaction mass (or related measured variables like torque on, or electrical power input to the stirring motor, etc.) along with corresponding measured values of the temperature, T(t), during bulk polymerizations as inputs to a model-based software sensor. Mankar et. al. (21,25-29) carried out bulk polymerization of MMA in a rheometer-reactor (viscometer) assembly under several temperature histories (isothermal, nonisothermal). They measured $\eta(t)$ (till onset of the gel effect) and T(t) online, and developed a correlation relating these variables to difficult-to-measure (online) quantities namely conversion (x_m) and Molecular weight (M_w). However, the range of applicability of this correlation is limited and there is a considerable scope for improvement. This work aims to provide sufficient experimental data [$\eta(t)$ and T(t)] generated under different temperature histories (step increase , step decrease) so that a correlation with wider range of applicability can be generated.

This work is presented in four chapters. Chapter 2 describes the experimental setup and polymerization procedure in vivid details. The results obtained from 22 experimental runs are presented in Chapter 3. Finally the conclusions drawn from the present work and recommendation for the future work are summarized in the Chapter 4.

CHAPTER 2

EXPERIMENTAL PROCEDURE

This chapter is divided into two sections, **Experimental Setup** and **Experimental Procedure**. In the first section vivid description of the setup is given, and in the next section the polymerization procedure is described in detail.

2.1 EXPERIMENTAL SETUP

The experimental system developed in the present study is shown schematically in Figure 2.1. The heart of the set-up is the rheometer-reactor assembly which is described below along with the control of temperature during the polymerization.

2.1.1 Rheometer-Reactor Assembly

A Haake® viscometer (M5 osc, Haake Mess-Technik GmbH, Germany) is modified to work as a rheometer-cum-reactor. A stainless steel cup-and-bob (Haake SV 2) is used for the measurement of viscosity. The rheometer-reactor assembly enables polymerization to be carried out in the annular gap between the cup and bob under almost any desired temperature history (step increase, step decrease, or continuously varying), while simultaneously measuring the viscosity *on-line*. The rheometer-reactor assembly was placed in a pressure vessel, as beyond a temperature of about 62^0 C, vaporization became significant and bubble formation started. These bubbles get entrapped in the reaction mass at high viscosities and lead to significant fluctuations in data on viscosity.

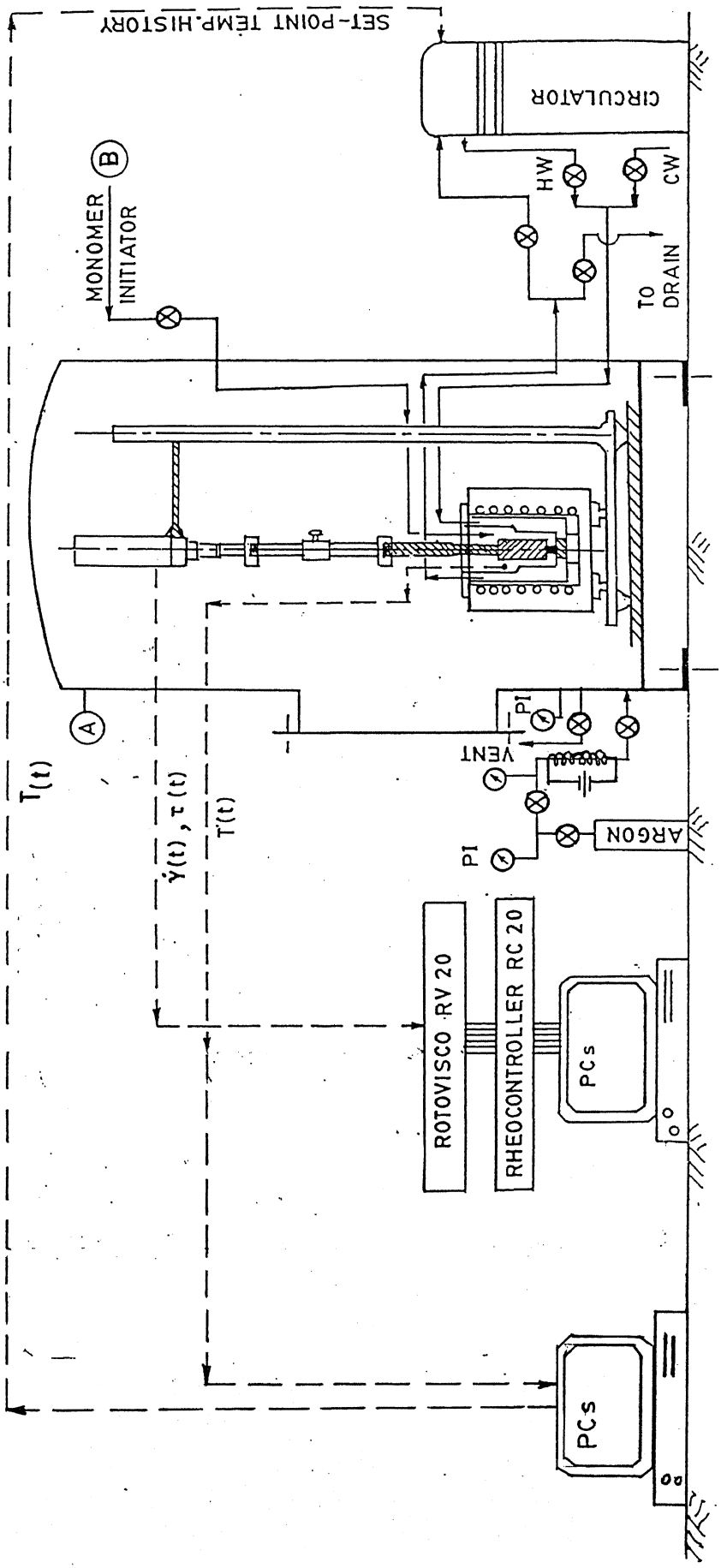


Figure 2.1 Schematic diagram of the experimental setup

The set-up is also equipped with a separate degassing unit, shown in Figure 2.2. It consists of a Stainless Steel vessel equipped with a sparging system to remove dissolved oxygen from the reaction mixture (monomer + initiator). It also serves as a feed vessel to the rheometer-reactor assembly which sends signals to a measuring system (Haake RV20) interfaced with a PC (Pentium-II) over a RS-232 serial line through the rheocontroller (Haake RC20). The viscosity of the reaction mass is measured at desired sampling times. The computer records the values of the shear stress, τ, and the shear rate, $\dot{\gamma}$, which in turn, are used to calculate the viscosity of the reaction mass as a function of time. The original cup (SV 2) supplied by Haake, was not provided with cooling jacket. It was necessary to modify the cup to include a jacket through which water at a desired temperature could be circulated (26) from the Julabo F10- MH (Julabo Labortechnik GmbH, Germany) circulating constant temperature bath. The details off the modified cup-and-bob assembly are shown in Figure 2.3. This new cup and bob has the same cup (inner) diameter as the Haake SV 2 cup. The dimensions of cup-and-bob are given in Table 2.1.

Table 2.1 Dimensions of Cup-and-Bob, SV 2 System

Inner Cylinder (Bob)	
Radius, R_i (mm)	10.1
Height,L (mm)	19.6
Outer Cylinder (Cup)	
Radius R_a (mm)	11.55

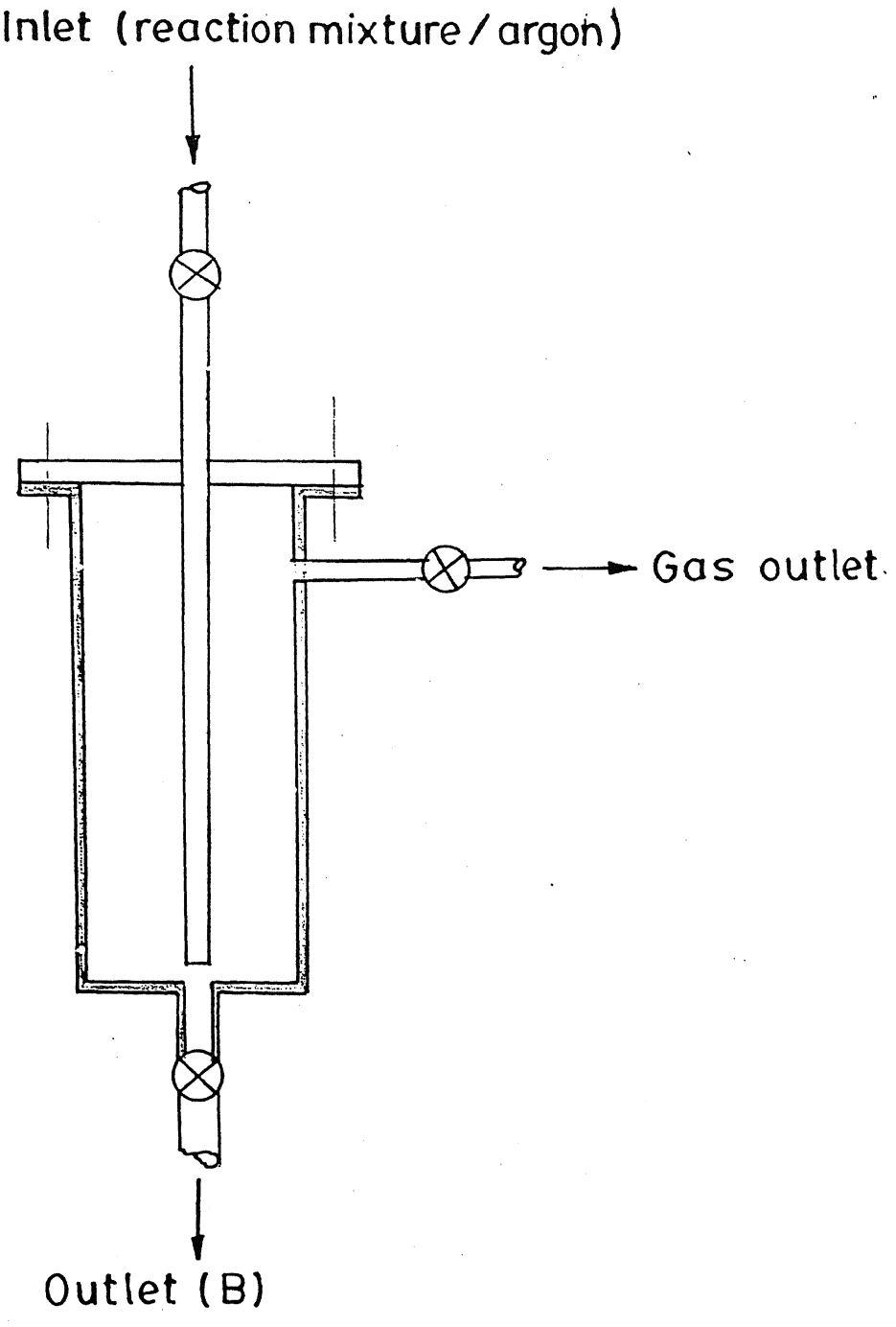


Figure 2.2 Schematic diagram of the degassing unit

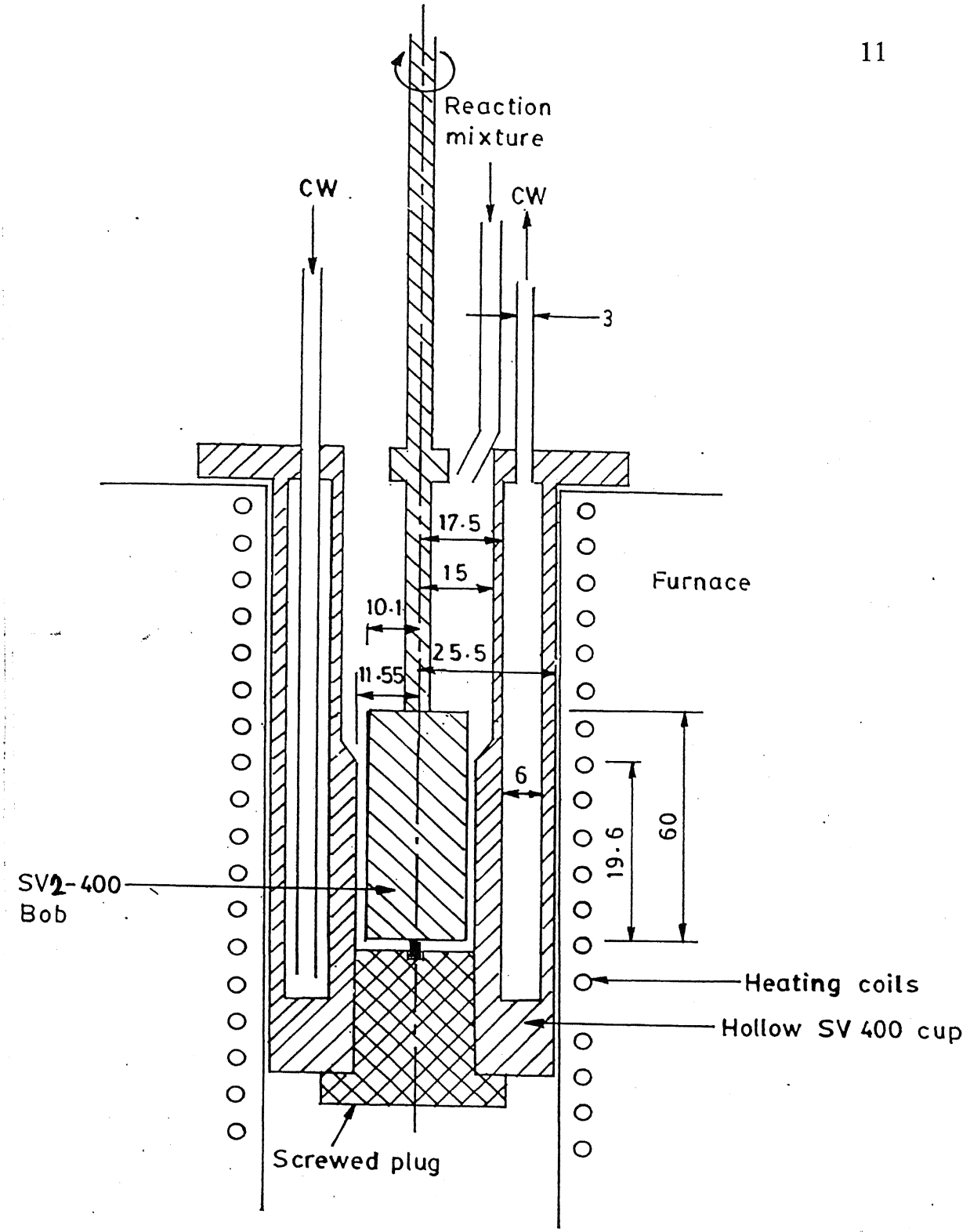


Figure 2.3 Schematic diagram showing details of modified cup-and-bob assembly used as rheometer-reactor. All dimesions are in mm (not to scale)

2.1.2 Calibration of Rheometer-Reactor Assembly

The rheometer-reactor assembly was calibrated prior to its use. Standard viscosity test fluids [E6000 (η = 5 Pa s) and E40000 (η = 43.3 Pa s) at 20^0 C], supplied by Gebrueder Haake GmbH, Germany were used for this purpose. The viscosity test fluid was filled in the cup and the temperature was lowered to 20^0 C ($\pm$ 0.2^0 C) and maintained thereafter by circulating cold water from a Julabo bath. The rheocontroller was set in the 'rotation' mode and following the standard procedures described in the operating manual calibration factors were determined. The calibration factors are given in Table 2.2.

Table 2.2 Calibration Factors for SV 2 System, Operating Mode - Rotation

Shear stress factor, A (Pa / % τ)	37.63
Shear rate factor, M (s^{-1} / % $\dot{\gamma}$)	5.34

where,

% τ = displayed shear stress

% $\dot{\gamma}$ = preset shear rate

2.1.3 Temperature Control

In this study different temperature histories, step increase and step decrease were implemented (within a variation of +/- 0.5 °C) by circulating water from constant temperature bath (Julabo). The temperature of the reaction mass was measured with a Pt100 sensor. The Julabo is also connected to a PC (Pentium-4) through RS232 serial connection, which facilitates recording temperature data continuously at given interval of time. In the Julabo bath there is an inbuilt PID type controller which controls the temperature of the water in the

ath. However, in the present setup we wish to control the temperature of the reaction mass in ıe annulus of the viscometer, which is lower than the temperature of the circulating water ırough the jacket. One way to overcome this problem is to control the bath temperature omewhat above the desired reaction temperature so that the reaction can be carried out at the lesired level. The reaction mass temperature can be monitored with a different sensor (Pt100 n the present case). There is option in the Julabo to make the external Pt100 signal as control nput which is expected to provide better control of the temperature of the reaction mass, but lue to some technical difficulties this option could not be used in this study. The set point for he bath which is required to maintain a particular temperature within the cup was found by ısing glycerol (1000-1500 cP) in the cup. 25 ml to 30 ml of glycerol was taken in the cup with the Pt100 sensor placed within it, and inside temperature of the cup was calibrated ıgainst Julabo. The calibration curve is shown in the Figure. 2.4.

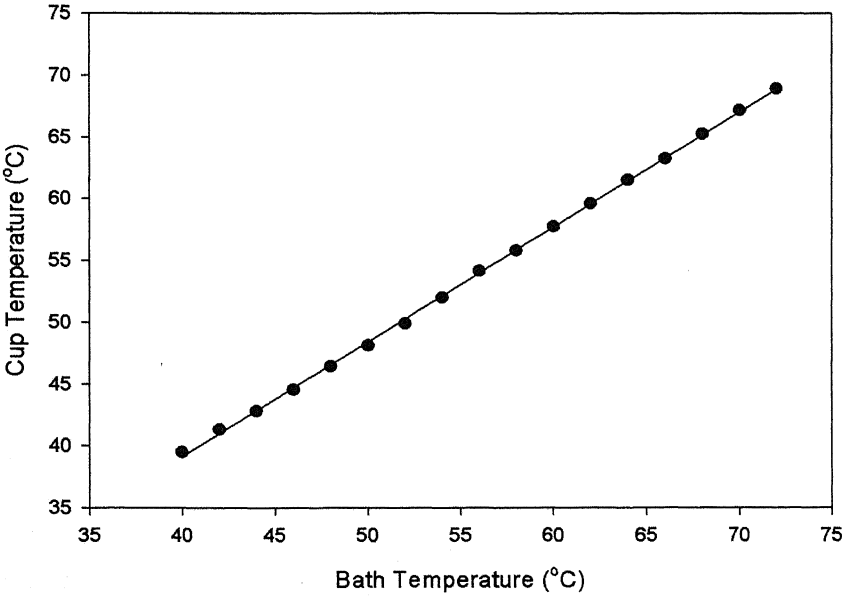


Figure 2.4 Calibration curve for the cup temperature against Julabo bath

'he implementation of the temperature step increase was achieved easily. It takes 8-10 min to ıcrease the temperature by 10°C. The temperature step decrease was a bit tricky since it took 5-20 min for the single stage cooling compressor in Julabo to lower the bath temperature by 0°C. To overcome this problem, just after set point change, circulation of the Julabo was topped and about 1.5 liters of the hot water was taken out from the bath, followed by the ddition of same amount of chilled water at 14°C. This was continued till the temperature of he bath falls at least 10°C below the set point, and then the circulator was started again. This mproved the cooling time and it was possible to attain the step decrease in 6-8 min.

nitially both the working sensor (bath sensor) and external sensor (Pt100 sensor) were :alibrated following the procedure described in the Julabo manual with the help of a fine livision (1/10 of a deg) thermometer. This is one point calibration to check the any bias that nay have developed. This calibration was checked prior to every run.

2.2 EXPERIMENTAL PROCEDURE

This section describes the polymerization procedure starting from purification of nonomer and initiator to washing of cup and bob after the polymerization.

2.2.1. Purification of Monomer and Initiator

LR grade MMA (Central Drug House, Mumbai, India) was purified prior to use. The monomer was washed three times with equal volumes of 5% sodium hydroxide (E. Merck, Mumbai, India) solution in double distilled water to remove the phenolic stabilizer [quinol (hydroquinone), 0.01%] present in it. The mixture was allowed to settle in a separating funnel forming two layers, the bottom inorganic layer was discarded leaving the monomer. Traces of sodium hydroxide were removed from the treated monomer by washing it thrice with double

stilled water. Settling times of at least two hours after each NaOH and water wash were lowed. The residual water was removed from the monomer by passing through beds of silica :l (mesh 3-8, NICE, Kochi, India) and molecular sieves (type 30-541, Linde division, Union arbide, Danbury, CT, USA). Fresh batches of regenerated silica gel and molecular sieves (by ashing and drying in an oven at about 70^0 C for 5 h and stored in air tight containers) were sed for every 250 ml batch of monomer. The monomer thus obtained was distilled under acuum (~ 50 mm Hg) at about 30^0 C. The distillation setup is shown in Figure 2.5. Ice cold 'ater was circulated in the condenser. A few beads of molecular sieves were added in istillation flask to augment flashing of monomer under vacuum. The distilled monomer was ept in a refrigerator. The final yield of monomer was about 60% of the original volume.

The initiator, LR grade 2, 2'- azoisobutyronitrile, AIBN (SAS Chemicals, Mumbai, ndia), was recrystallized from LR grade methanol (Ranbaxy Laboratories, S. A. S. Nagar, 'unjab, India). A saturated solution of AIBN was prepared (at room temperature) in a conical lask. The solution was filtered using ordinary filter paper. The filtrate was chilled in a efrigerator to crystallize the AIBN. The crystals were recovered by filtration and dried in an ven under vacuum at room temperature to avoid thermal decomposition. The mother liquor vas stored for future use. The procedures followed are well documented in the literature 30,31).

2.2.2 Polymerization Procedure

The cup and bob assembly is put in place, after through cleaning, inside the pressure vessel. The annular gap between cup and bob of viscometer is very small (~ 3 m.m.), hence it s importanat to ensure that the cup and bob are concentric. So before starting the experiment

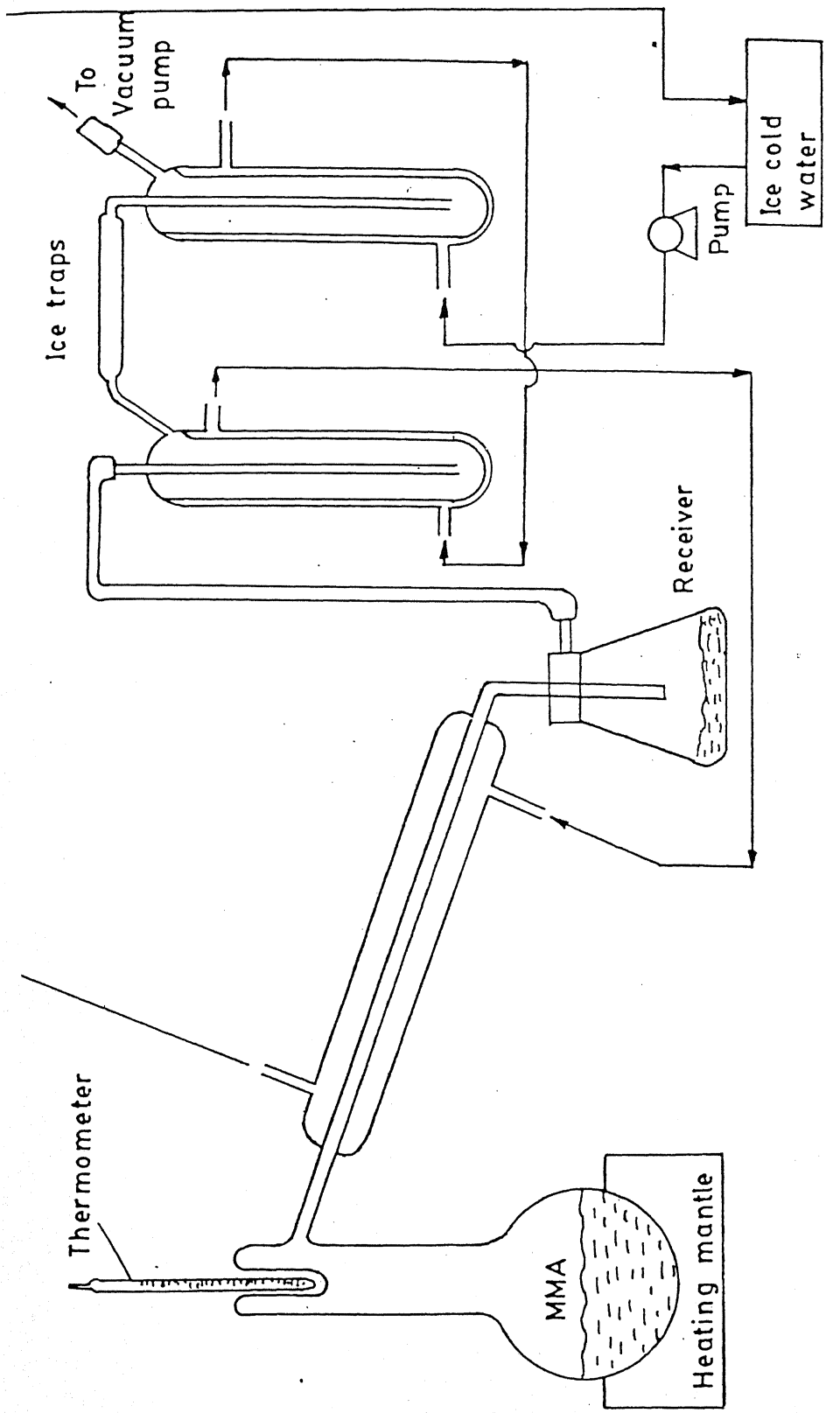


Figure 2.5 Vacuum distillation setup for monomer

the alignment of the cup and bob was checked at no load condition (empty cup) by setting a shear rate for a particular time, and it is given in Table 2.3

Table 2.3 Shear Rate Settings in Haake Software at no Load Condition, Operating Mode – Rotation

Shear rate (s^{-1})	Time (min)
10	0
10	2

At this setting the shear stress value should be within the range 0 -0.1 Pa, which ensures the proper alignment of the cup and bob. After alignment was ensured the, the Pt100 was put into the cup and its tip was fixed just above the bob. The lid of the pressure housing was then tightly bolted to the flange provided in the setup, to make it leak proof. The pressure housing was then flushed with preheated (using heating tape wrapped on the copper tubing) Argon from a compressed gas cylinder for at least 5 to 7 min. The preheating was necessary, as sudden expansion of the gas would lower the temperature of the reaction mass.

Desired amount of initiator is weighed correctly up to four decimal places, and taken in a sampling bottle, followed by the addition of 30 ml of monomer to it. It was then taken in the degassing unit. This degassing unit was washed with LR grade acetone (Ranbaxy Laboratories, S. A. S. Nagar, Punjab, India) before every use. Argon (IOLAR-I grade) was sparged at a very slow rate for about 10 minutes to remove the dissolved oxygen (a reaction inhibitor). The degassing unit was then sealed. The oxygen-free reaction mixture was then transferred (by gravity) from the degassing unit directly into the annular gap of the rheometer-reactor assembly preheated to the desired temperature. As soon as the reaction mixture was

ısferred, the Haake software with desired shear rate setting (see Appendix B) was started. ɜ reaction mixture attained the desired temperature within 2-3 minutes after which a ıstant temperature (within an error of $\pm$ 0.5^0 C) was maintained. Argon was then charged in pressure vessel and a pressure of around 2.5 Kg/cm^2 was maintained until the end of the ı. After that the temperature was monitored continuously and set point was changed within 0.5 °C as required to maintain the temperature in the reaction mass.

ɜual inspection of polymer in the rheometer gap at the end of polymerization confirmed ɜence of bubbles. The exothermic heat generated at the onset of the gel effect was removed circulating water at ambient temperature through the jacket of the cup for a very short ɾiod of time around that period. No further cooling was required till the end of the run. rque measurements were started at a shear rate of $10s^{-1}$. The shear rate was lowered to2 s^{-1} ıen the viscosity of the reaction mixture reached a value above 2 Pa.s. The shear rate was ther lowered to 0.1 s^{-1}at any point of time between 3 Pa.s. to 30 Pa.s. The shear rate setting given in detail in the Appendix 2. These ensured that the values of viscosity were close to ɜ zero-shear values. The measuring system got overloaded above viscosities of about 40,000 s and the bob automatically stopped rotating to avoid damage to the system. The rotating b worked as a stirrer during the polymerization. The shear rate, shear stress and temperature ɜre recorded by the PC (Pentium –II) as a function of time.

2.3 Washing of Cup and Bob

After polymerization the screw in the lower part of the bob was disassembled and the ıole assembly was put in a beaker filled with dichloromethane (Qualigens, Bombay, India) d the beaker was place in the refrigerator. For initial washing dichloromethane from evious wash was used. After 3-4 hours when the polymers became a little soft the Pt100

was taken out from it, and the bob was also pushed out from the cup using a screw feed, which make the dissolution of polymer very fast. After all the polymer was dissolved, the cup and bob assembly was placed in fresh dichloromethane. After every wash dichloromethane was kept for future use. Finally before putting the cup and bob into the pressure housing, it was washed with LR grade acetone (Ranbaxy Laboratories, S. A. S. Nagar, Punjab, India).

CHAPTER 3

RESULTS AND DISCUSSION

The main thrust of this work is to generate experimental data on viscosity of the reaction mass during bulk polymerization of MMA under different temperature histories (step increase, step decrease). These data can be used for developing improved viscosity correlations.

Bulk polymerization of MMA was carried out in the rheometer-reactor assembly at two different initiator (AIBN) loadings (I_0 = 15.48 and 25.80 mol/m^3) and at four different temperature histories (50°C to 60°C, 60°C to 70°C, 70°C to 60°C and 60°C to 50°C). The initiator loadings were the same as those in the study of Balke and Hamielec (30) carried out under isothermal conditions in small glass ampoules. As the reaction proceeds, the reaction mixture becomes more and more viscous. The viscosity of the reaction mass increases dramatically after the onset of the gel effect, when x_m as well as M_w, increase sharply. We were able to obtain several data points for the viscosity of the reaction mass in the gel effect region using sampling times (at which viscosities were recorded by the PC) varying from 84 s to 6 s (the sampling times were chosen somewhat arbitrarily, but were larger than the response times for viscosity measurements in all cases). Data were obtained till reasonably high values of viscosity were reached, well into the gel effect region.

Before conducting the step change experimental runs, two isothermal polymerization were carried out (at 70°C for I_0 = 15.48 mol/m^3 and at 60°C for I_0 = 25.8 mol/m^3). The viscosity vs time data are shown in Figure 3.1 and 3.2 respectively, along with the temperature history in Figure 3.3. Also included in Figure 3.1 and 3.2 are data from Mankar (29). A good reproducibility ensure proper working of the viscometer.

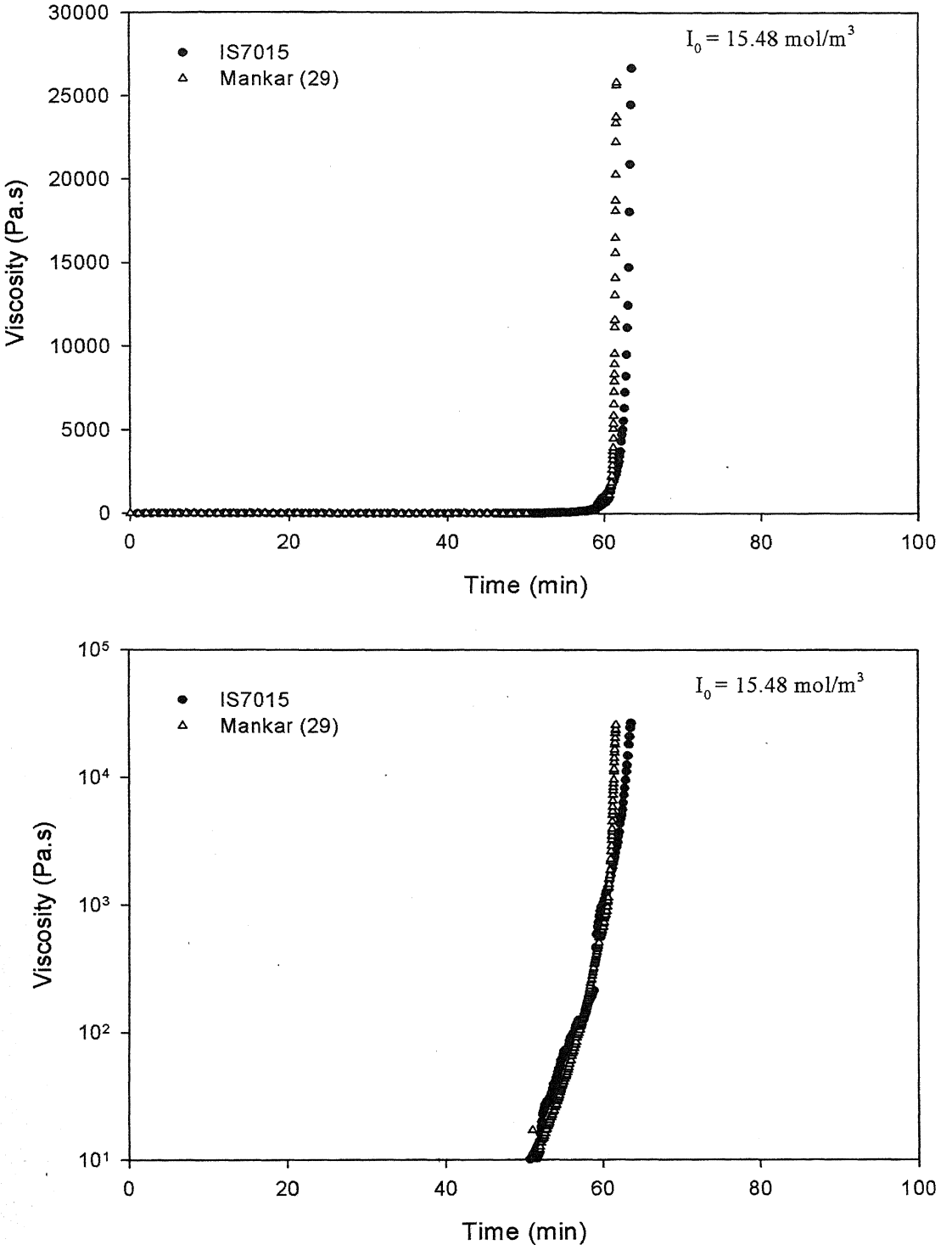


Figure 3.1 Experimental data on viscosity vs. time during isothermal polymerization at 70°C

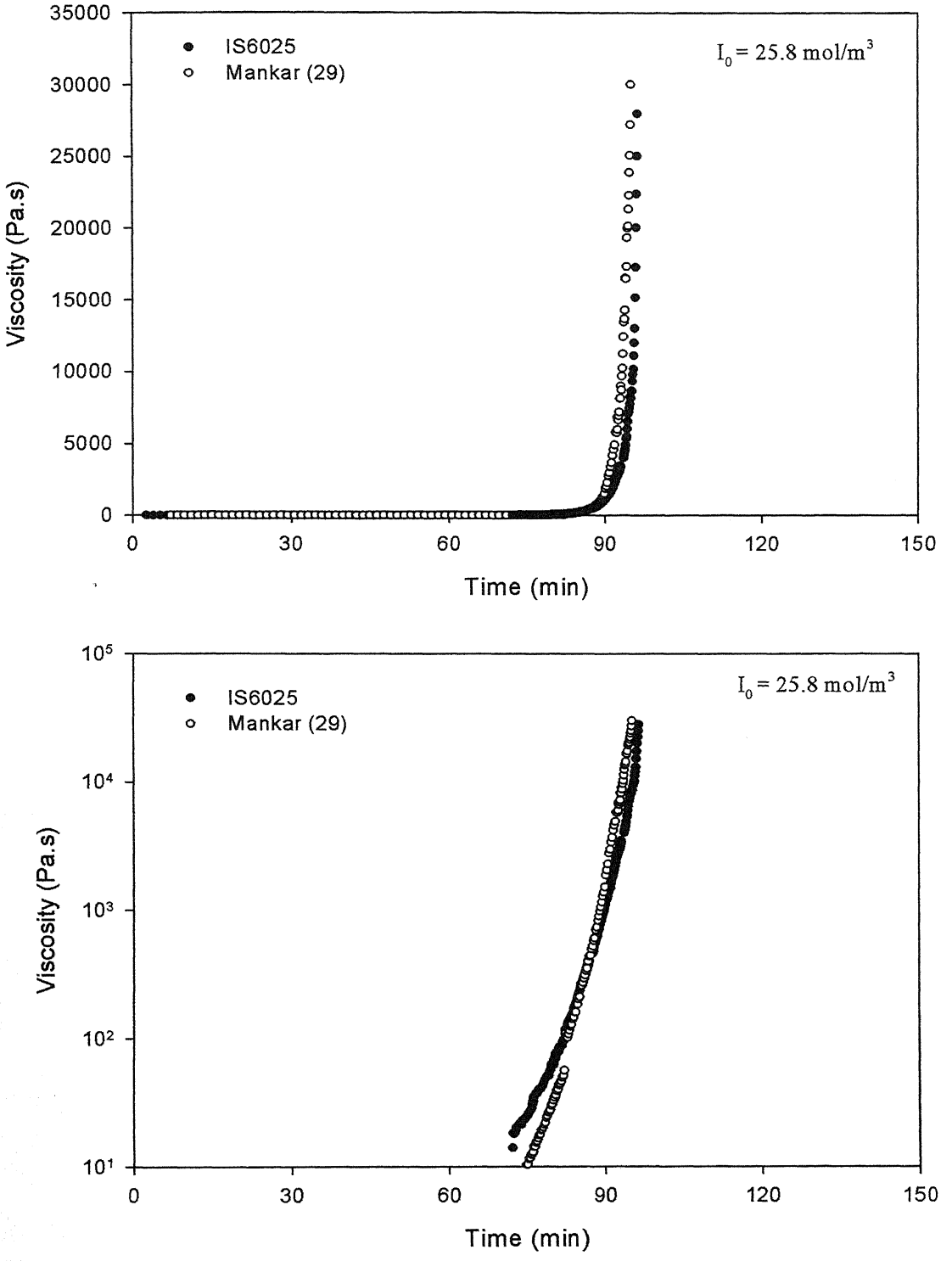


Figure 3.2 Experimental data on viscosity vs. time during isothermal polymerization at 60°C

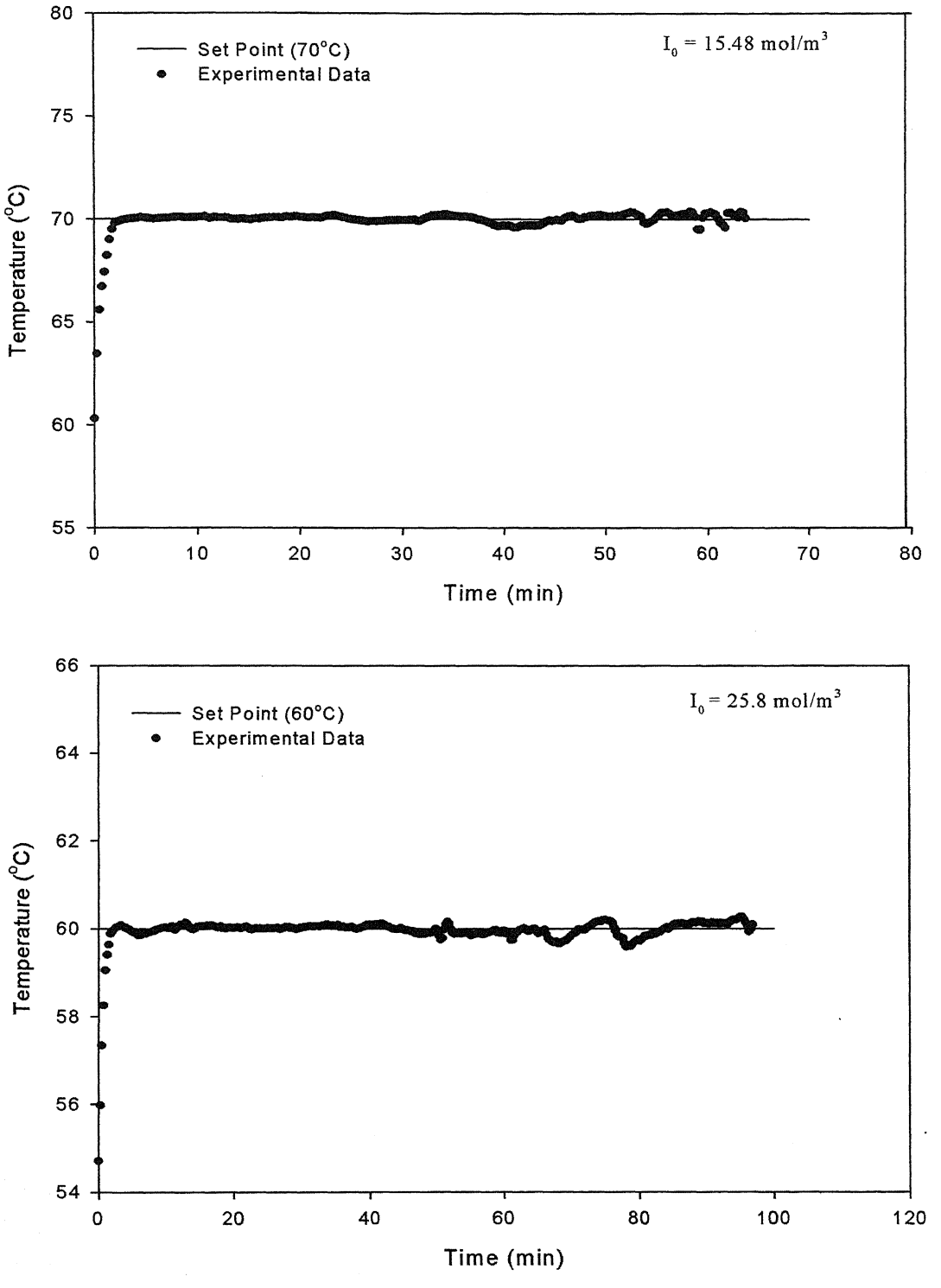


Figure 3.3 Temperature histories for isothermal 70°C and isothermal 60°C polymerizations

series of experimental runs under different temperature histories, step increase and step crease, were conducted (see Appendix A). The results (viscosity vs. time) are presented ng with the isothermal data of Mankar (29) for comparison. For example, data of step rease from 50°C to 60°C are presented along with isothermal 50°C and isothermal 60°C a. This was done to have a feeling of the qualitative validity of the generated data. plicate runs were taken for every type of temperature step change, to ensure the roducibility of the data. The viscosity vs. time data so generated are plotted on linear scale well as semi-log scale (viscosity in log scale). Near the gel region the increase of viscosity exponential in nature, and hence the semi-log plot gives a near straight line with a certain pe, which represent rate of increase of viscosity. This slope is steaper at higher nperature, since at higher temperature, the rate of increase of viscosity is higher than that of ower temperature.

e experimental runs were assigned numbers which represented the operating conditions. r isothermal runs first two letters IS represents isothermal conditions. Next two digits note the temperature in °C and the last two digits give initiator loading. For rest of the runs st letter S is for step test, second for increase or decrease (I or D) in temperature, third letter for initiator loading (B ≡ 15.48 mol/m^3 an C ≡ 25.8 mol/m^3), the next two digits provide the tial and final temperature of the step change (56 means step increase from 50°C to 60°C .) and the last 2 or 3 digits give time in minutes at which the step change was given. A efix letter R is added to indicate that it is a replicate run.

1 B SERIES EXPERIMENTS

Ten runs in this series were conducted, including two replicate run with initiator loading of 15.48 mol/m^3.

3.1.1 Run No. SIB56100 and SIB56150

In these two runs a step increase of temperature from 50°C to 60°C was given at two different times; 100 min and 150 min respectively, after the start of the runs. The step changes were given much before the gel region, because once the reaction goes into the gel region it takes almost no time to complete. The viscosity vs. time data for these two runs are shown in Figure 3.4 along with the data of replicate run of SIB56100 (RSIB56100) and Figure 3.5 presents the temperature histories, both set point and experimental, for the two runs. The attainment of gel region for step change at 100 min is earlier than that for 150 min. As expected, in the higher viscosity range the curves in semi-log plot for both the cases are almost parallel to the isothermal 60°C data. A small jump in the viscosity data in semi-log plot is due to the fact that as viscosity of the reaction mass increases, the shear rate setting has to be changed. During the change which takes almost 30 sec to 60 sec no measurements are available which results in a jump in semi-log plot. This jump is not visible in the linear plot as it is in the low viscosity region (200 Pa.s.).

3.1.2 Run No. SIB6740 and SIB6760

A step change from 60°C to 70°C was given at two different times, 40 min and 60 min respectively after the start of the run. The time of giving step change was selected on the basis of attainment of gel region for isothermal 60°C run from the data of Mankar (29). The viscosity vs time data for these two runs are shown in Figure 3.6, and corresponding temperature histories are shown in Figure 3.7. The run with step change at 40 min attained gel region before the step change at 60 min. In the semi-log plot initially up to a small range of viscosity the step change curves are almost parallel to isothermal 60°C data, and in the higher

viscosity range the lines are parallel to the isothermal 70°C data. Initially when the temperature was kept at 60°C, the rate of increase of viscosity was similar to isothermal 60°C run and at the later stage the rate of increase of viscosity become similar to isothermal 70°C. The temperature rise in the case of step change at 60 min towards the end (see Fig. 3.7) is due to the insufficient removal of the excessive exothermic heat generated in the gel region.

3.1.3 Run No. SDB7615 and SDB7625

In this two runs we gave a step decrease in temperature was given from 70°C to 60°C at two different times, 15 min and 25 min respectively from the start of the runs. Again the times were selected on the basis of the approaching time of the gel region for isothermal 70°C run. The viscosity vs time data are shown in Figure 3.8 and temperature histories for both the runs are shown in Figure 3.9. Step change at 25 min run attained gel region before the other one as expected. A viscosity value above 28,000 Pa.s. for step change at 15 min and above 27,000 for the other one were recorded. The initial fluctuations in the viscosity data are in very low range of viscosity compared to what we get finally, and can be ignored. Initially the viscosity data for step changes in semi-log plot are almost parallel to 70°C data, and finally both the data become parallel to 60°C data, as expected. During step increase tests it was noticed that rise of 10°C temperature took only 7 to 8 minutes, but cooling is a slower process and unless externally cooled, Julabo needed 10 minutes or more to lower the temperature by 10°C. For both the runs temperature at the final stage takes off a little bit from the set point due to the exothermic heat generation in the gel region, and with a shot of cooling water through the jacket it is kept within +/- 0.5 °C of the set point. If cooling shot is not used the temperature may rise up to 10°C to 15 °C above the set point.

3.1.4 Run No. SDB6530 and SDB6560

A step change from 60°C to 50°C was given at two different times, 30 min and 60 min respectively. The initiator loading was same as before (I_0 = 15.48 mol/m^3). These two times were selected on the basis of attainment of gel region for isothermal 60°C run. The viscosity vs time data for this two runs are shown in Figure 3.10 along with the data of replicate run of SDB6560 (RSDB6560) and temperature histories are shown in Figure 3.11. Near congruence of the replicate runs ensured the reproducibility of the data. Similar to previous case the run with step change at a later time (60 min) attains the gel region sooner the other one. We reached a high value of viscosity, above 40,000 Pa.s in the case of step change at 60 min, whereas for the other case a value of above 29,000 Pa.s was attained before the viscometer bob stopped rotating. The strain rate setting for high viscosity range is 0.1 s^{-1} but most of the cases the corresponding rate internally set was in the range of 0.11 to 0.14 s^{-1}. However in the case of the result SDB6560it was found to be 0.09 which led to a high viscosity of above 40,000 Pa.s at the limiting value of the shear stress. If were one to use a still lower value of say 0.05 s^{-1} the viscosity range of 70,000 Pa.s was reached. However, these data are npt reliable since at such a low strain rate (~ 0.05 s^{-1}) the viscosity was not stable and even though the shear rate continuously increases the viscosity fluctuates widely indicating the instability in the rate of strain. As expected the viscosity data are parallel to the isothermal 50°C in the high viscosity region. The jump in the viscosity data for step change at 60 min is somewhat large, and is due to the fact that it took a little extra time at that instance to change the shear rate setting. The temperature profiles for these two cases show no fluctuations at the end, because the exothermic heat generation in the case of isothermal 50°C is not much and could easily be controlled.

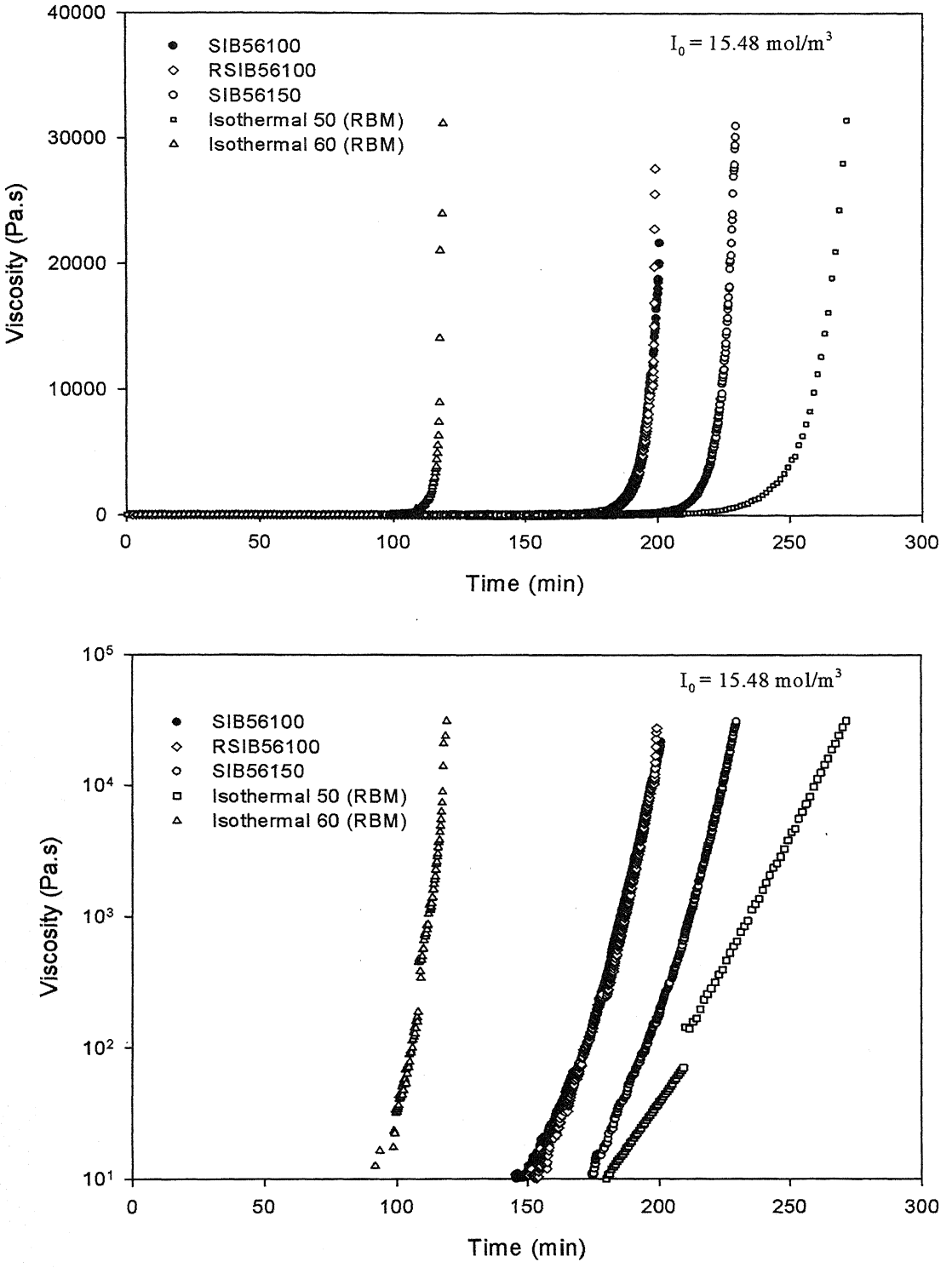


Figure 3.4 Experimental data on viscosity vs time for temperature step change from 50°C to 60°C at different times (100 min and 150 min). $I_0 = 15.48$ mol/m^3.

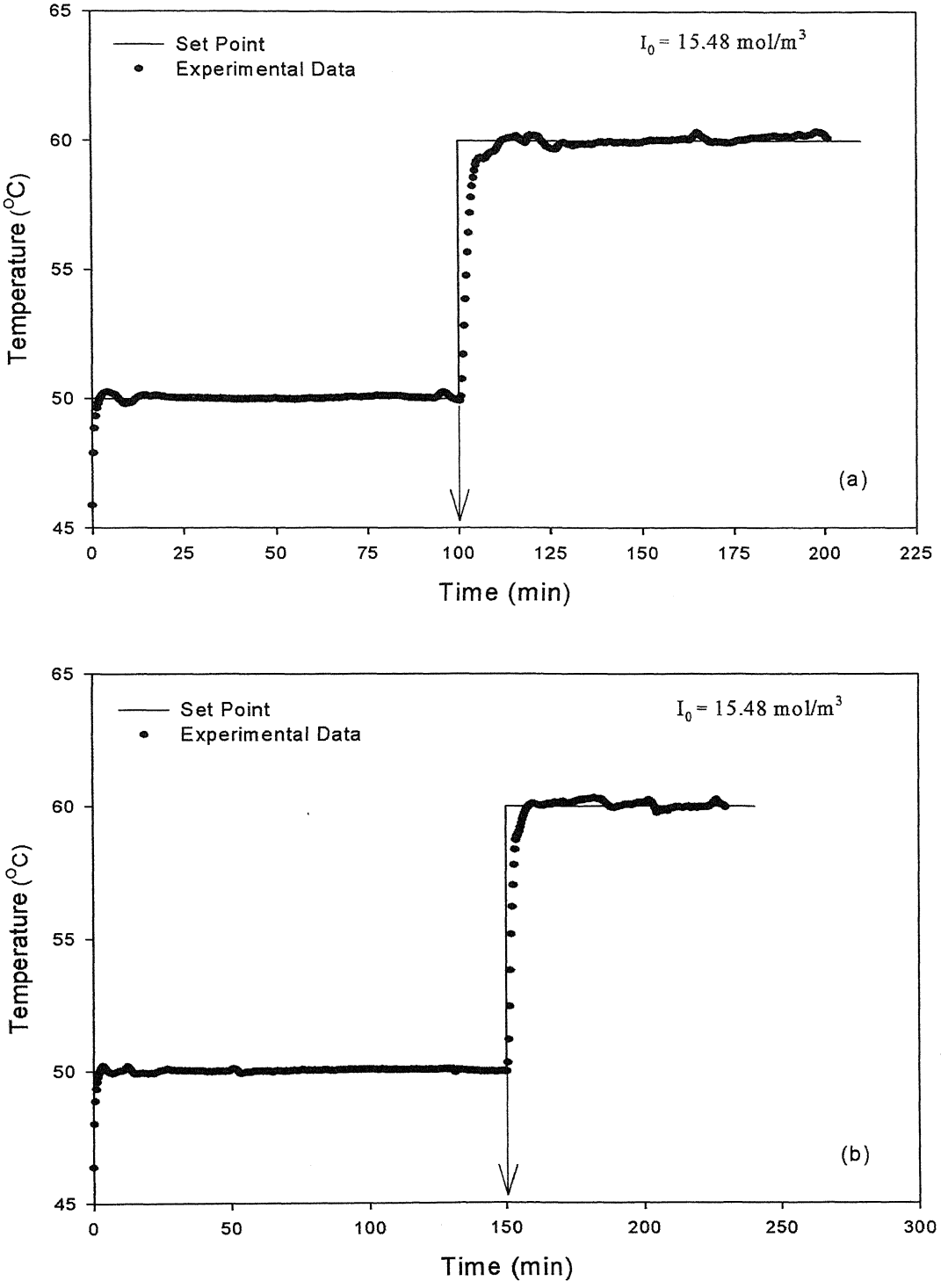
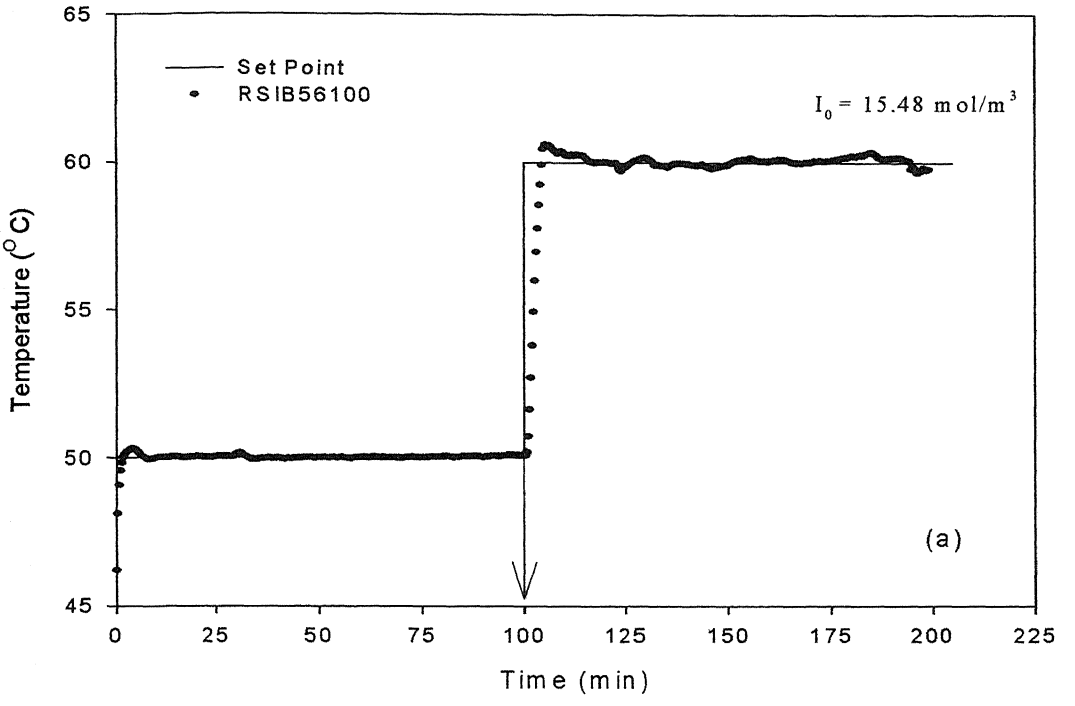
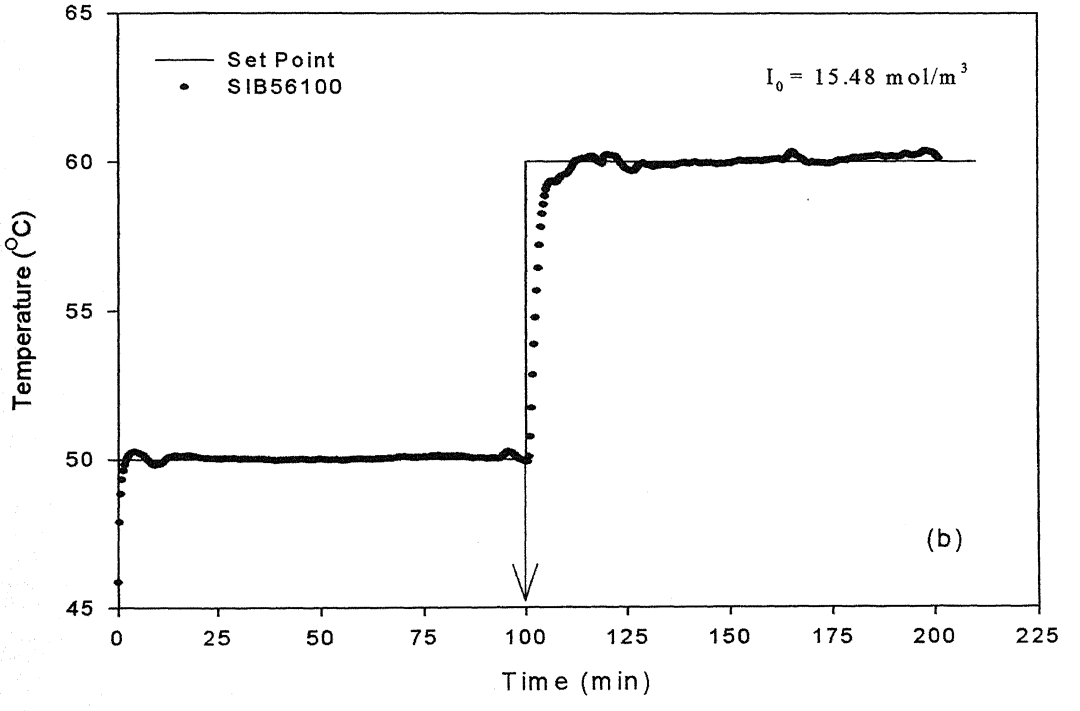
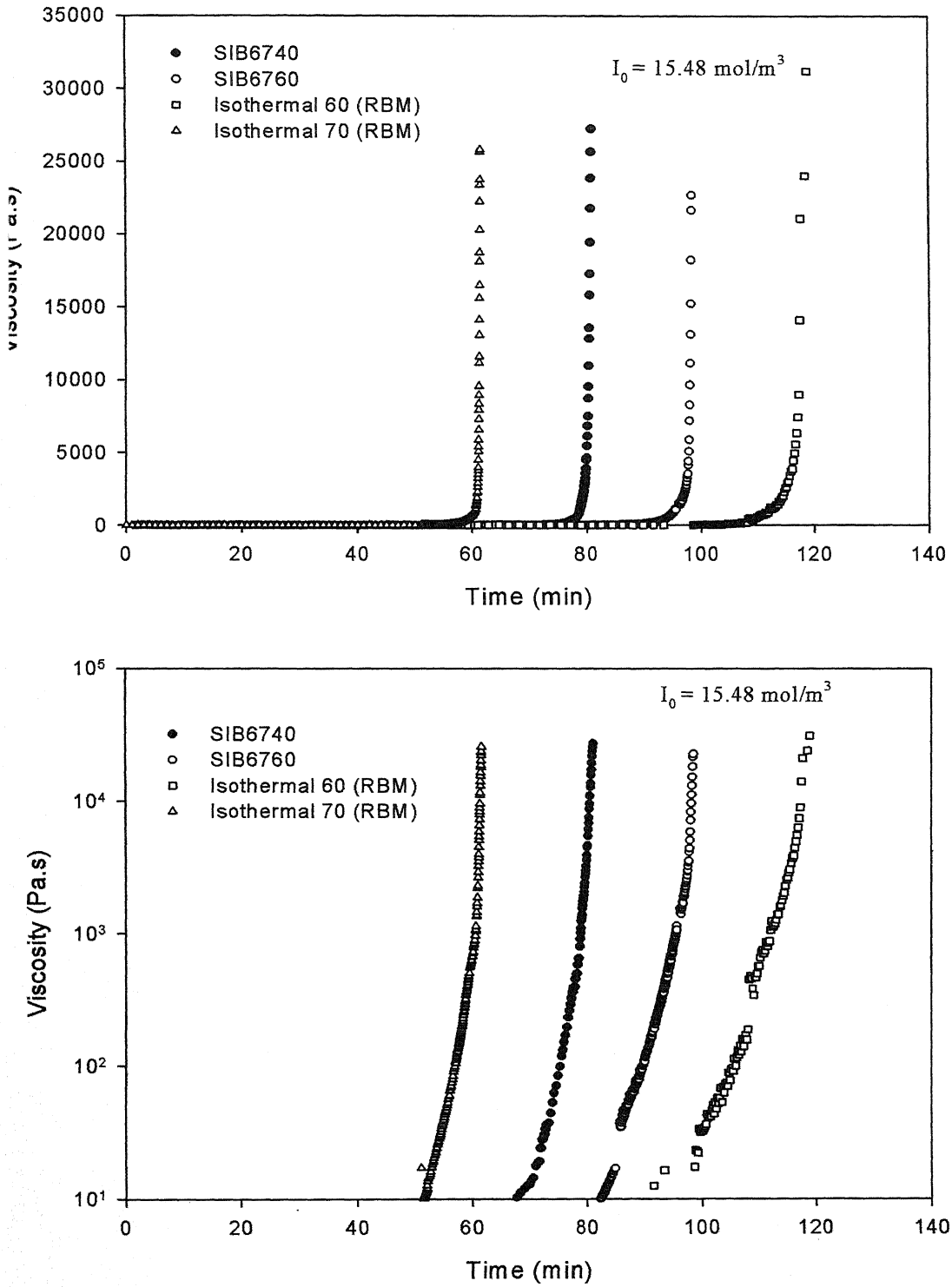


Figure 3.5 Temperature histories for step change from 50°C to 60°C at (a) 100 min and (b) 150 min.





ure 3.5.1 Temperature histories for step change from 50°C to 60°C at 100 min (a) replicate and (b) original



gure 3.6 Experimental data on viscosity vs time for temperature step change from 60°C to 70°C at different times (40 min and 60 min). $I_0 = 15.48$ mol/m^3.

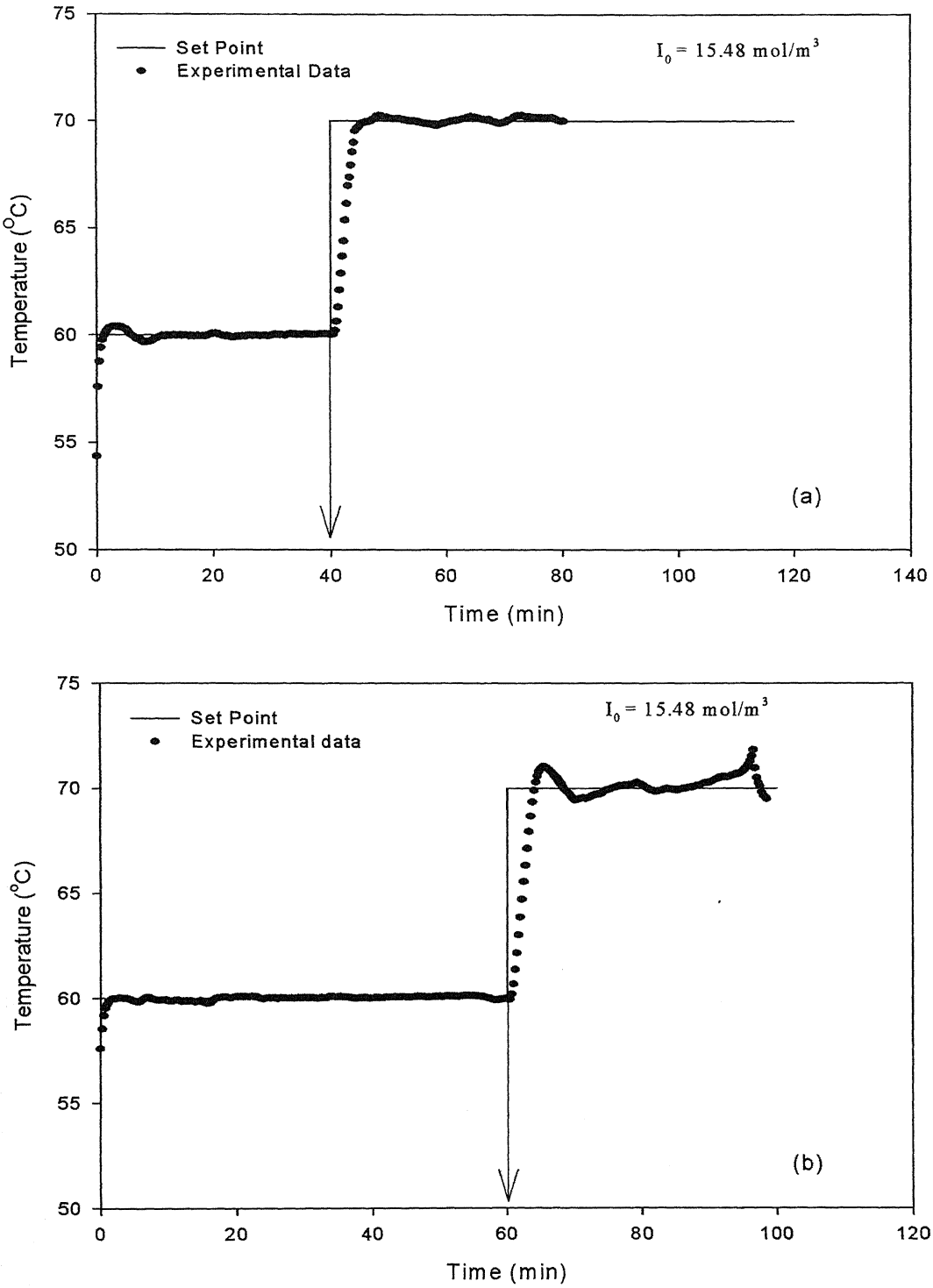
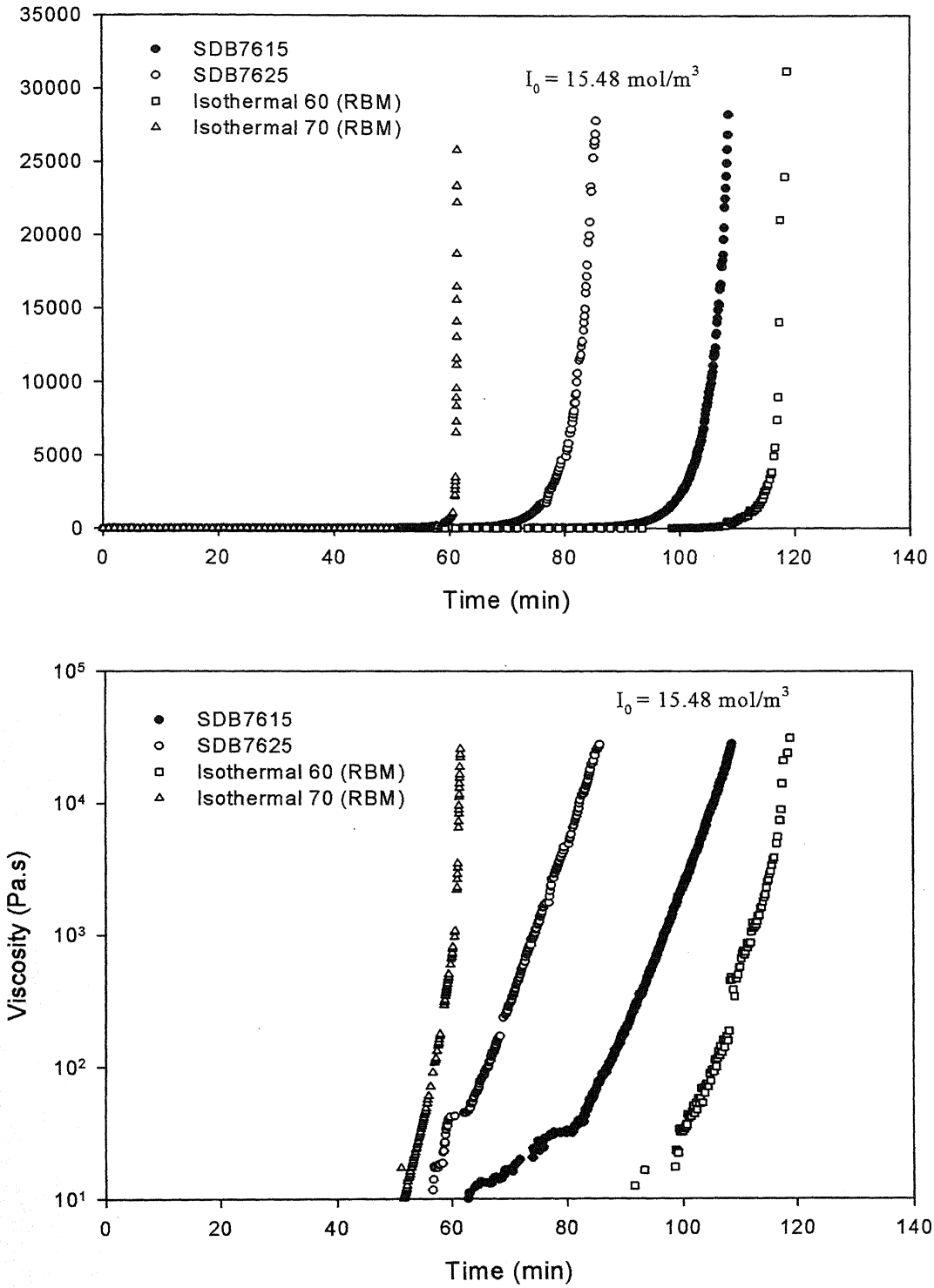


Figure 3.7 Temperature histories for step change from 60°C to 70°C at (a) 40 min and (b) 60 min.



ɡure 3.8 Experimental data on viscosity vs time for temperature step change from 70°C to 60°C at different times (15 min and 25 min). I_0 = 15.48 mol/m^3.

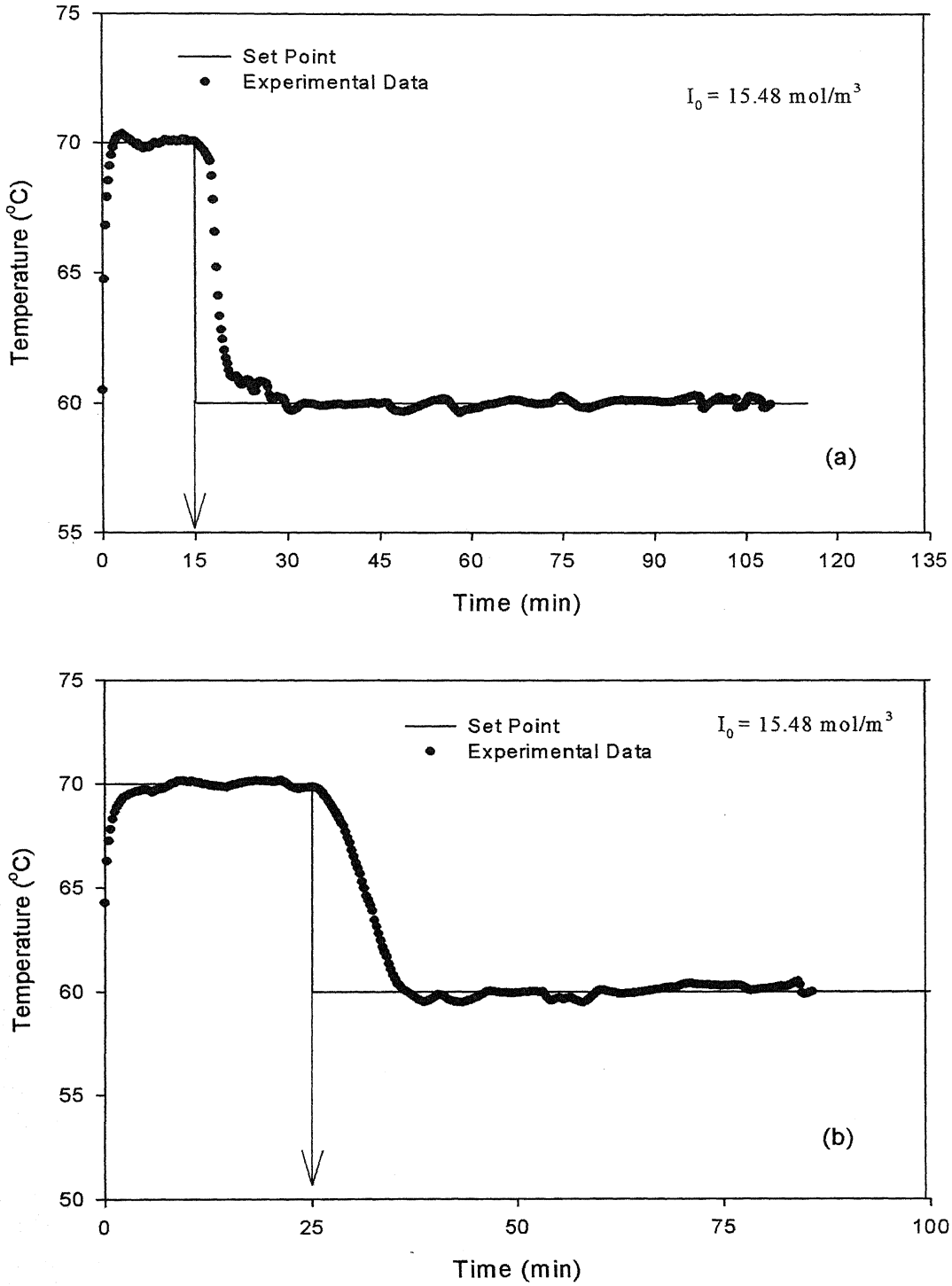
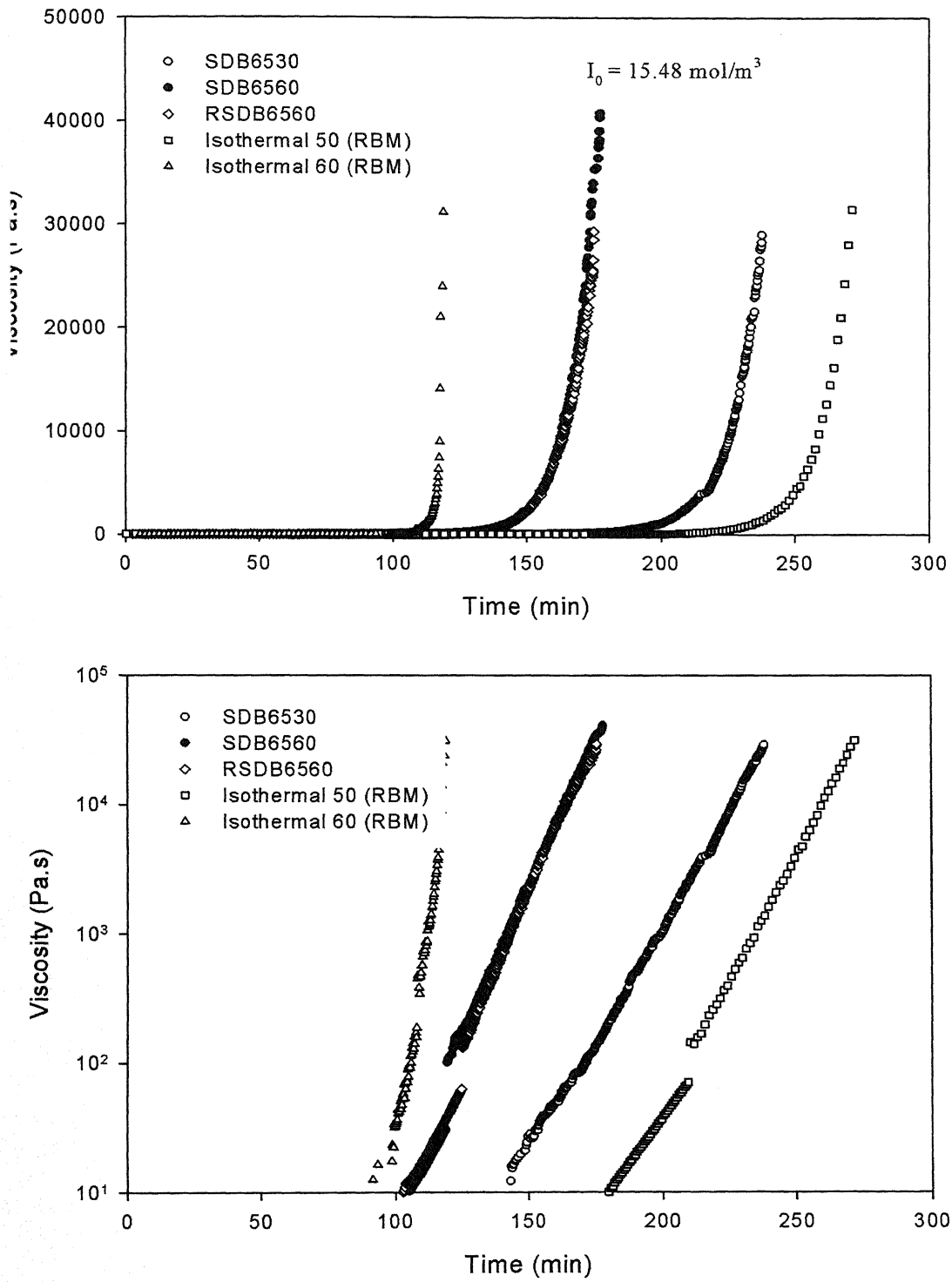


Figure 3.9 Temperature histories for step change from 70°C to 60°C at (a) 15 min and (b) 25 min.



gure 3.10 Experimental data on viscosity vs time for temperature step change from 60°C to 50°C at different times (30 min and 60 min). I_0 = 15.48 mol/m^3.

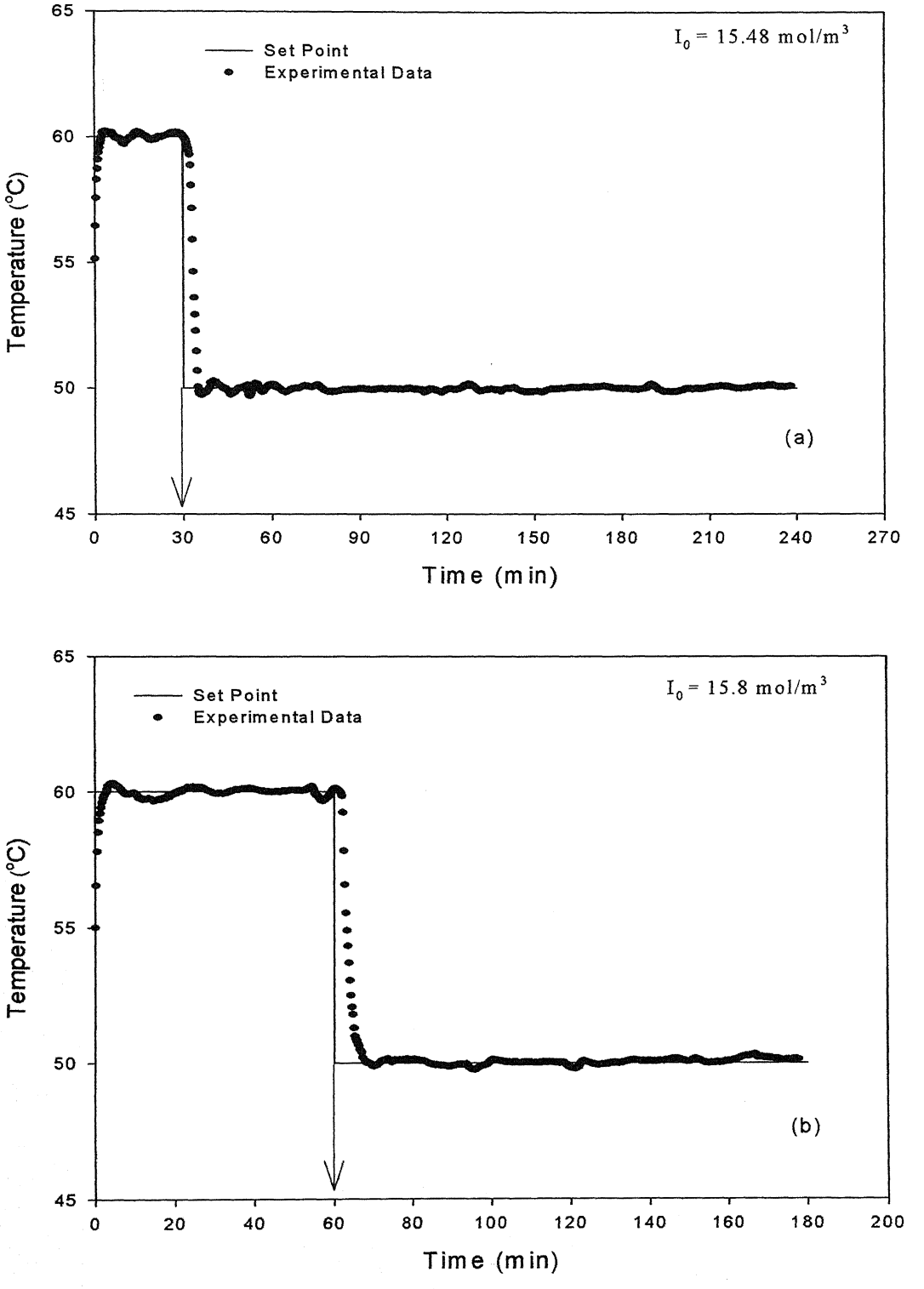
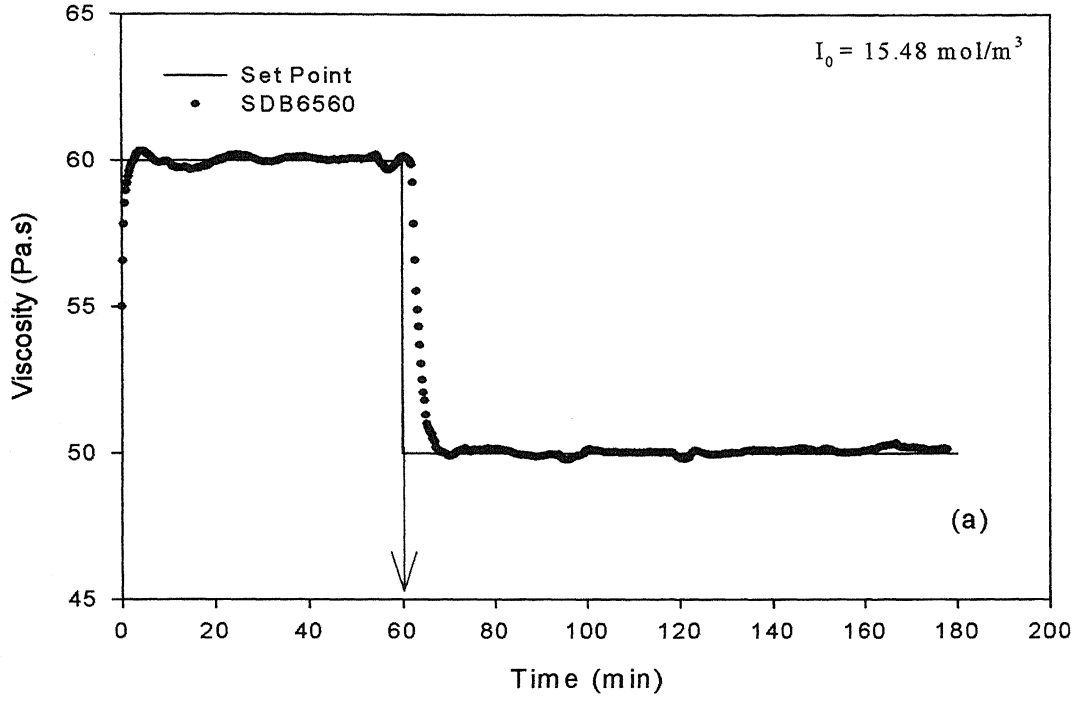
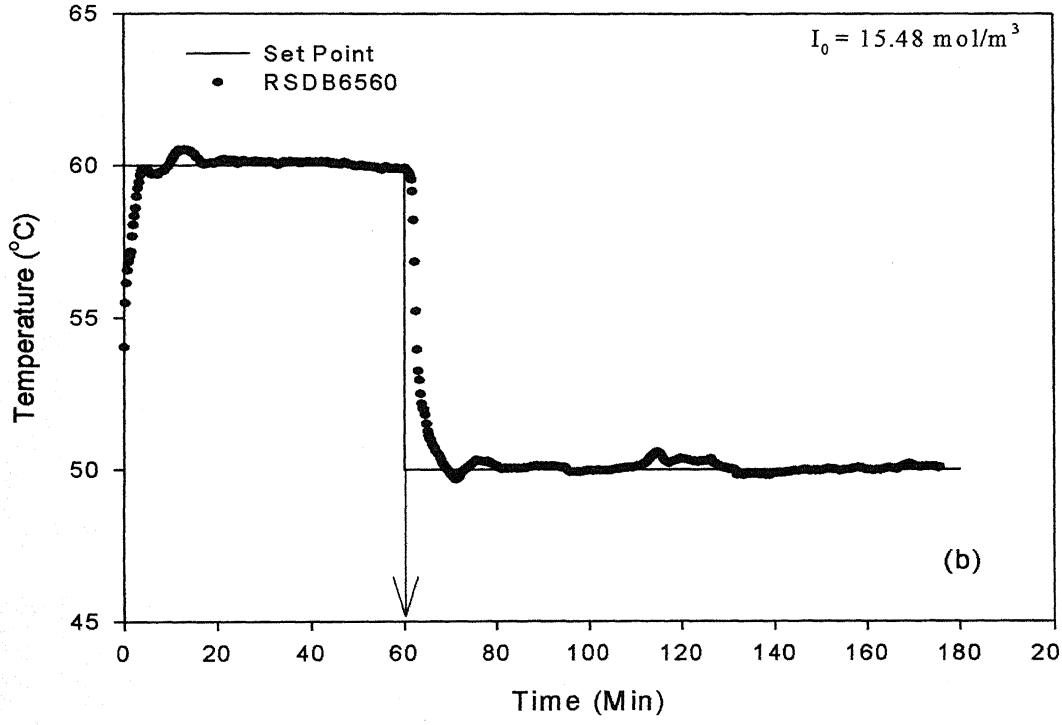


Figure 3.11 Temperature histories for step change from 60°C to 50°C at (a) 30 min and (b) 60 min.





igure 3.11.1 Temperature histories for step change from 60°C to 50°C at 60 min (a) original and (b) replicate

3.2 C SERIES EXPERIMENTS

The above eight runs with the same step increase and step decrease in temperature at similar times were repeated with the initiator loading of 25.8 mol/m^3. Two replicate runs for different type of step increase and step decrease were carried out. The results obtained are almost similar, but the time scale has been shifted towards the lower value, as expected.

3.2.1 Run No. SIC56100 and SIC56150

A step in temperature from 50°C to 60°C was given at two different times100 min and 150 min in two different runs. The viscosity vs. time data are shown in Figure 3.12, and temperature histories of this two runs are shown in Figure 3.13. As usual we reached a viscosity value of above 26,000 Pa.s. for SIC56100 and above 27,000 Pa.s. for SIC56150. As expected, gel region attainment of the run SIC56100 was achieved before SIC56150. For the run SIC56100, almost in the entire range, the viscosity curve in semi-log plot is parallel to the isothermal 60°C data. This is because the viscosity of the reaction mass increases appreciably only after giving the step change. For the run SIC56150 initially the data are almost parallel to the isothermal 50°C, and as the viscosity increases to a high value, the curve becomes parallel to the isothermal 60°C data, since at 150 min viscosity of the reaction mass had reached a significantly high value.

3.2.2 Run No. SIC6740 and SIC6760

For these two runs temperature was increased from 60°C to 70°C at two different times of 40 min and 60 min after the start of the run. The viscosity vs. time data are shown in Figure 3.14 along with the data of replicate run of SIC6740 (RSIC6740) and temperature histories are shown in Figure 3.15. On the semi-log plot, the viscosity data for SIC6740

rough out its whole range is almost parallel to the isothermal 70°C data, which is expected. : the lower range the viscosity data for SIC6760, is parallel to the isothermal 60°C data and the higher range it is almost parallel to the isothermal 70°C data. One noticeable thing in e semi-log plot is that there is no jump in the viscosity data for both the runs. This is due to e reason that for these two cases time required to complete the reaction is small and most of e viscosity rise occurs within last 10 minutes before which the shear rate setting was done. mperature for both the cases was found to rise towards the end because of excessive heat of action in the gel region which was controlled within +/- 0.5°C by giving a shot of cold water rough the jacket.

2.3 Run No. SDC7615 and SDC7625

The viscosity vs time data are shown in Figure 3.16 along with the data of the replicate n of SDC7615 and temperature histories are shown in Figure 3.17. The replicate data gives od match with the earlier one. The viscosity data for SDC7615 are parallel with the thermal 60°C data, as expected, and the data for SDC7625 are initially parallel to thermal 70°C and at higher viscosity value it become parallel with isothermal 60°C data.

2.4 Run No. SDC6530 and SDC6560

The viscosity vs. time data are shown in Figure 3.18 and temperature histories are own in Figure 3.19. The viscosity data for SDC6530 is through out its range is parallel with isothermal 50°C data, as expected. The viscosity data for SDC6560, at its initial stage, incide with isothermal 60°C, since in the first 60 minutes the viscosity rises to a nsiderable value After the step decrease in temperature is introduced, the polymerization ws down as indicated by decrease in slope of viscosity vs time curve.

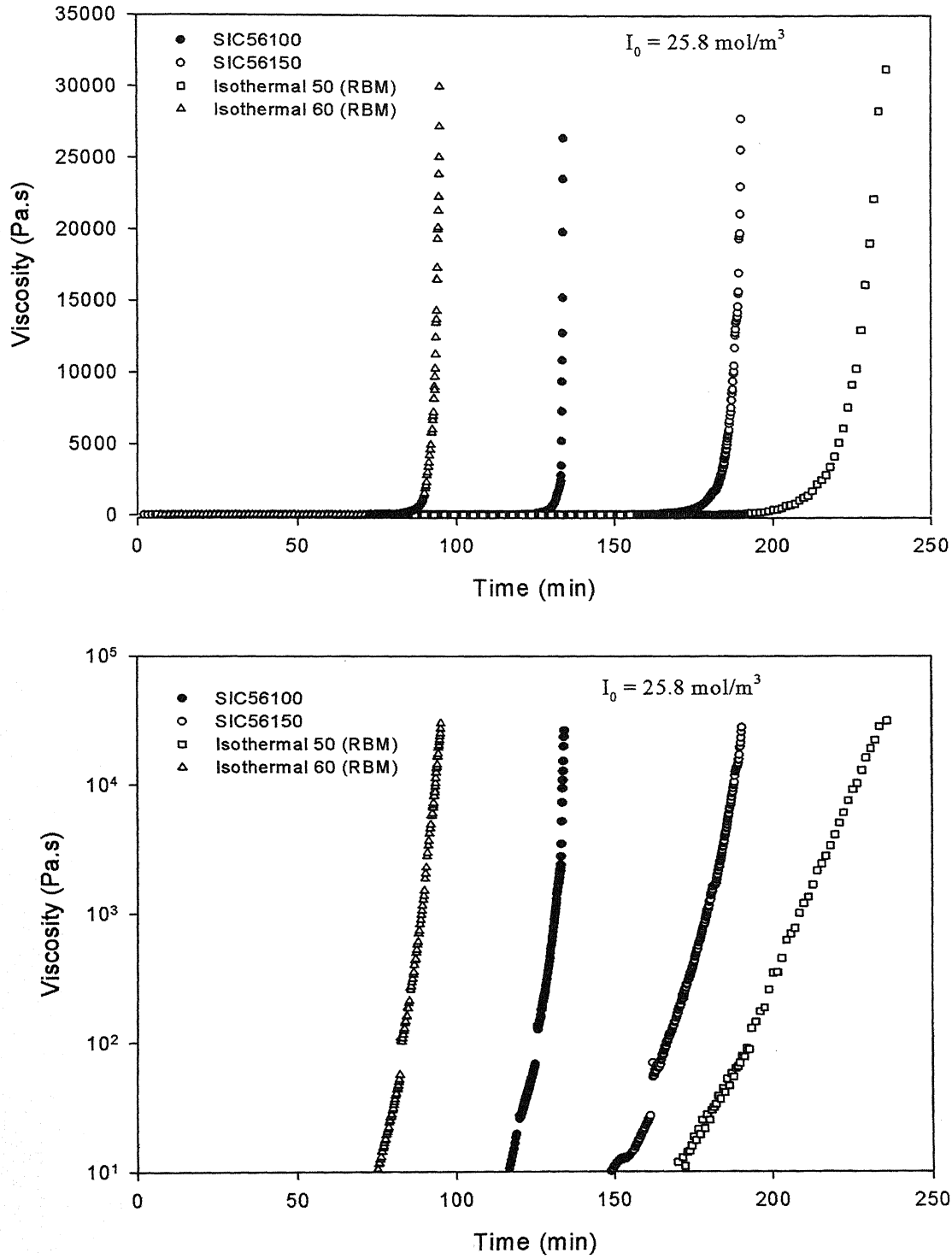
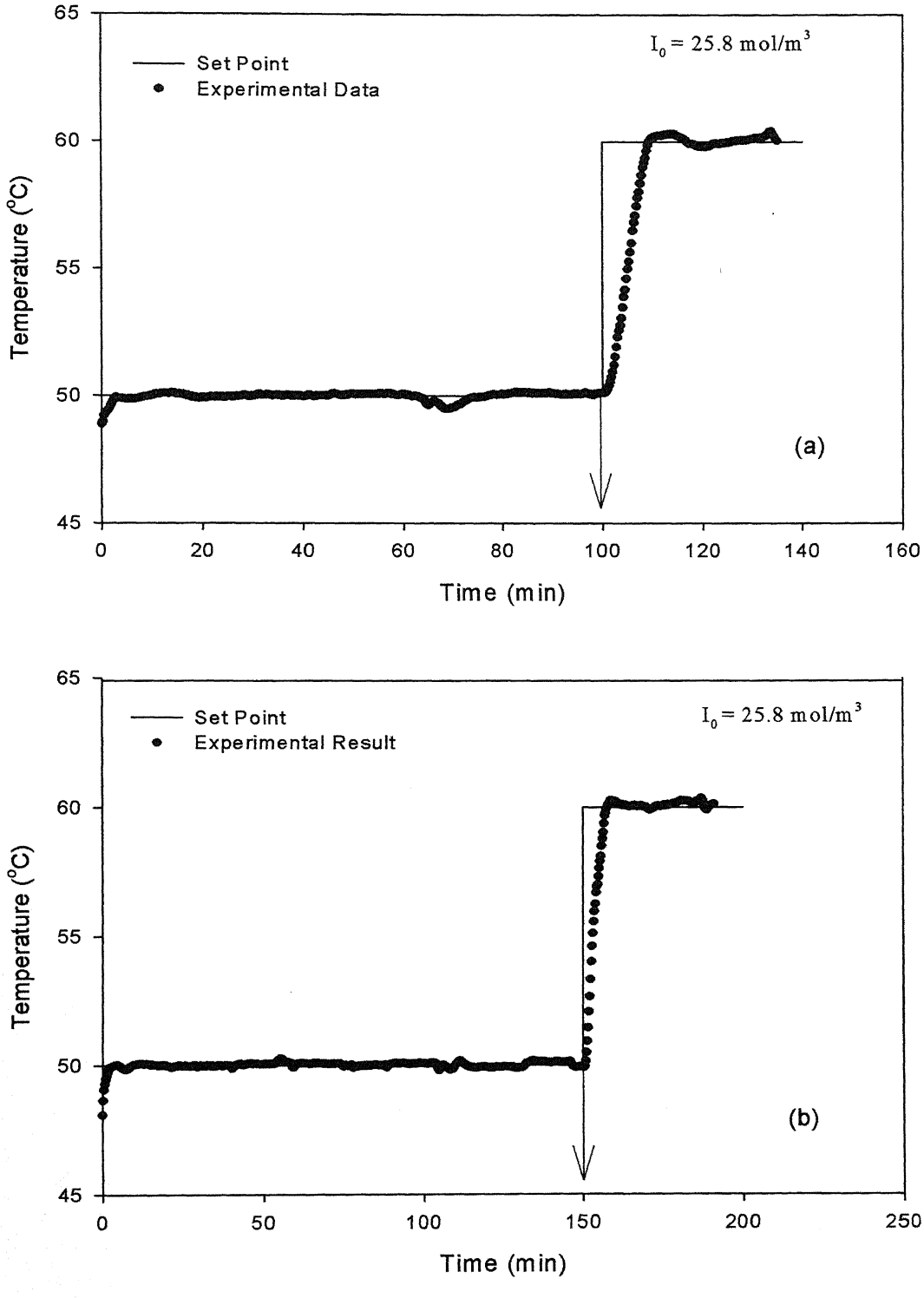


Figure 3.12 Experimental data on viscosity vs time for temperature step change from 50°C to 60°C at different times (100 min and 150 min). $I_0 = 25.8$ mol/m^3.



gure 3.13 Temperature histories for step change from 50°C to 60°C at (a) 100 min and (b) 150 min.

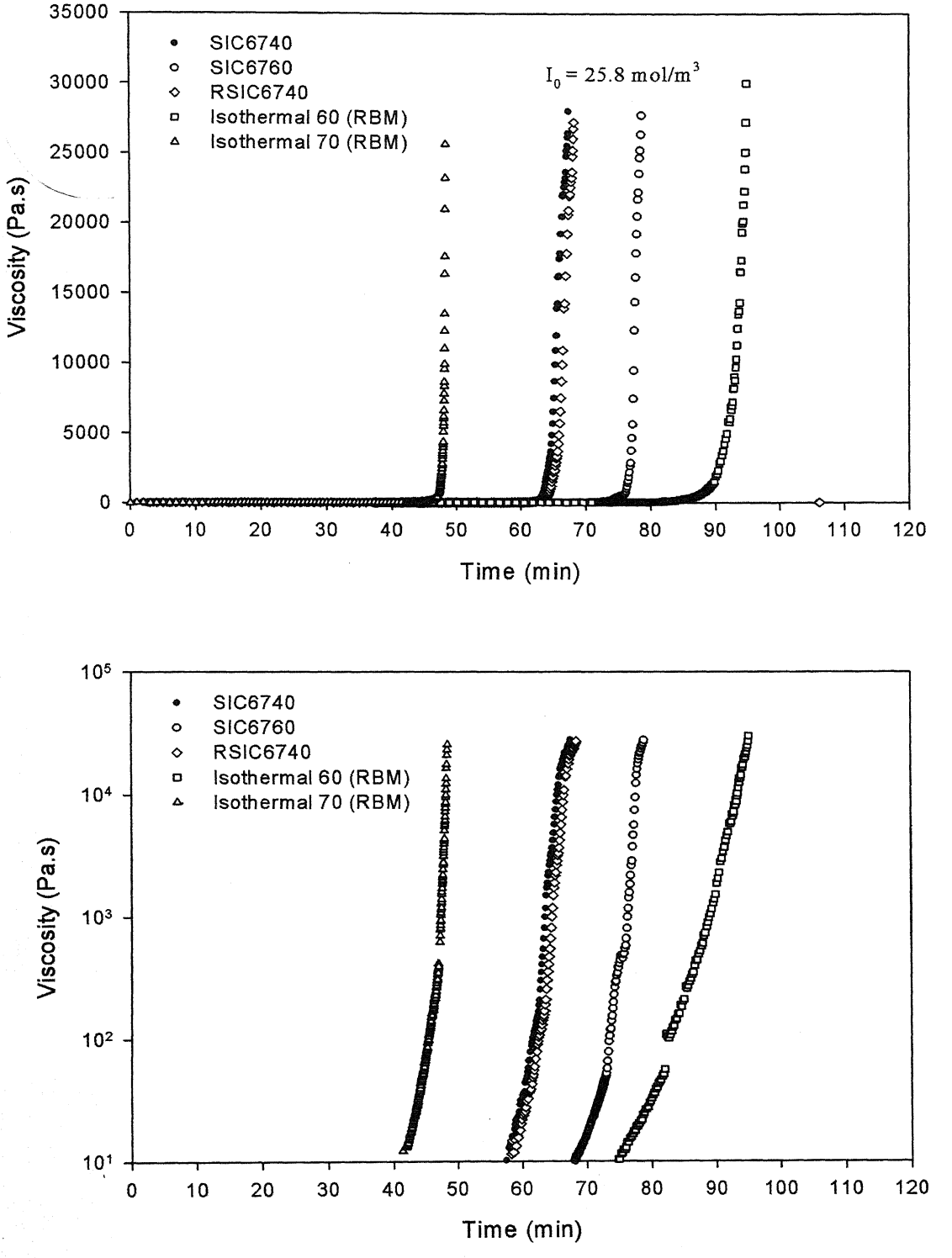


Figure 3.14 Experimental data on viscosity vs time for temperature step change from 60°C to 70°C at different times (40 min and 60 min). $I_0 = 25.8$ mol/m^3.

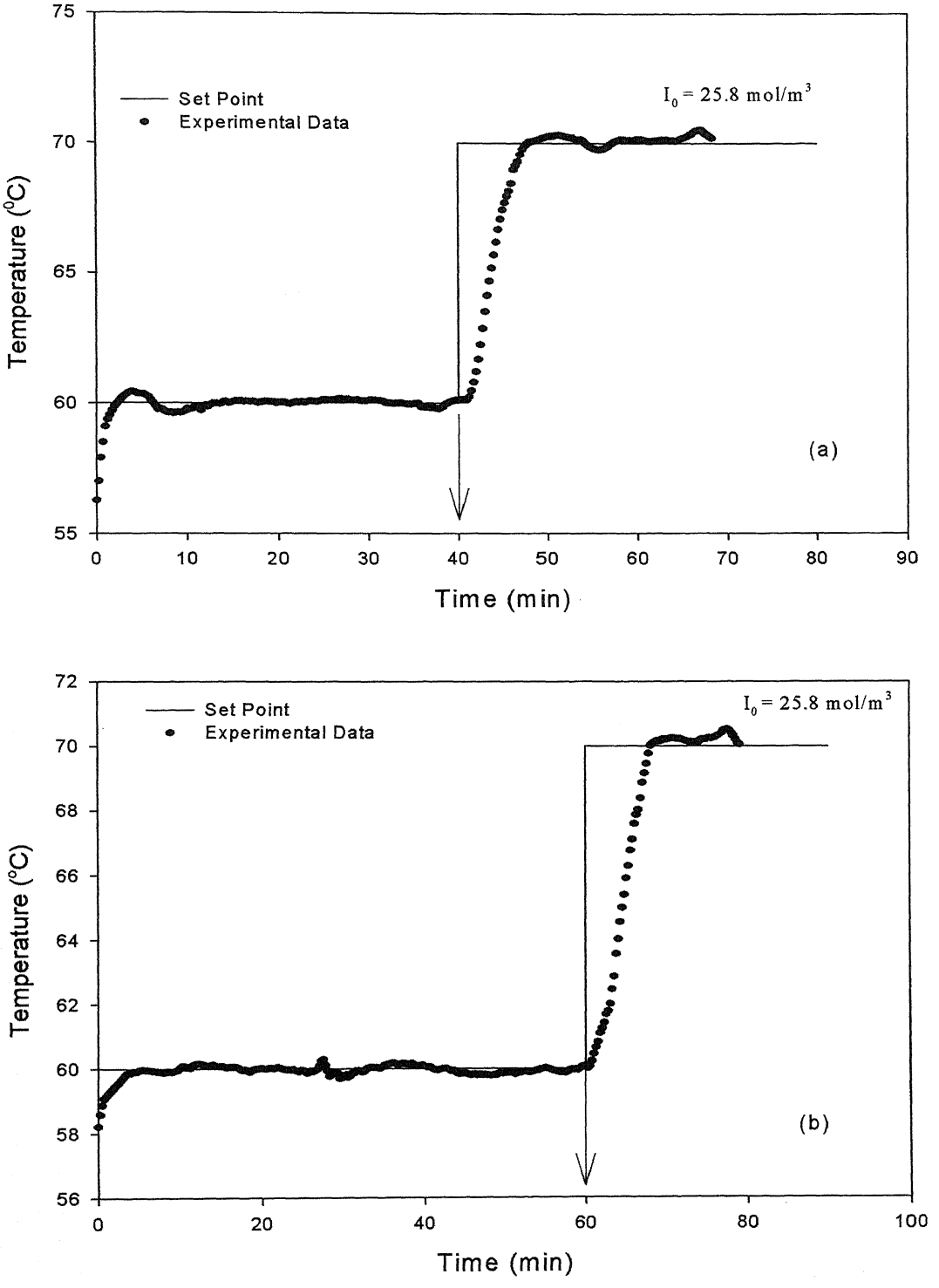
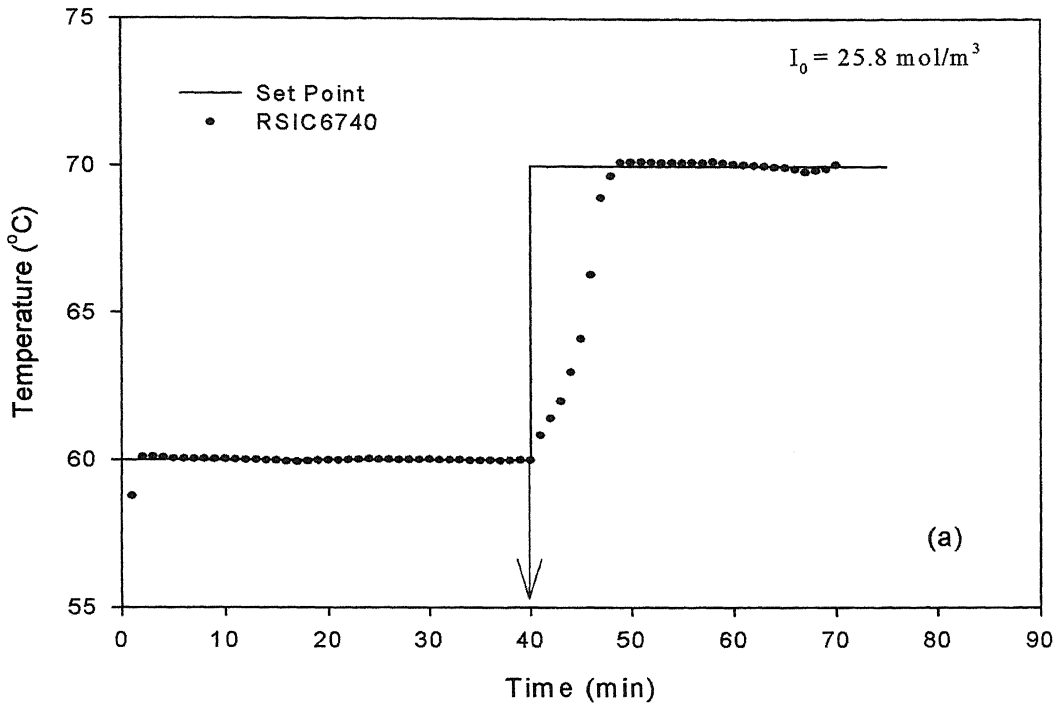


Figure 3.15 Temperature histories for step change from 60°C to 70°C at (a) 40 min and (b) 60 min.



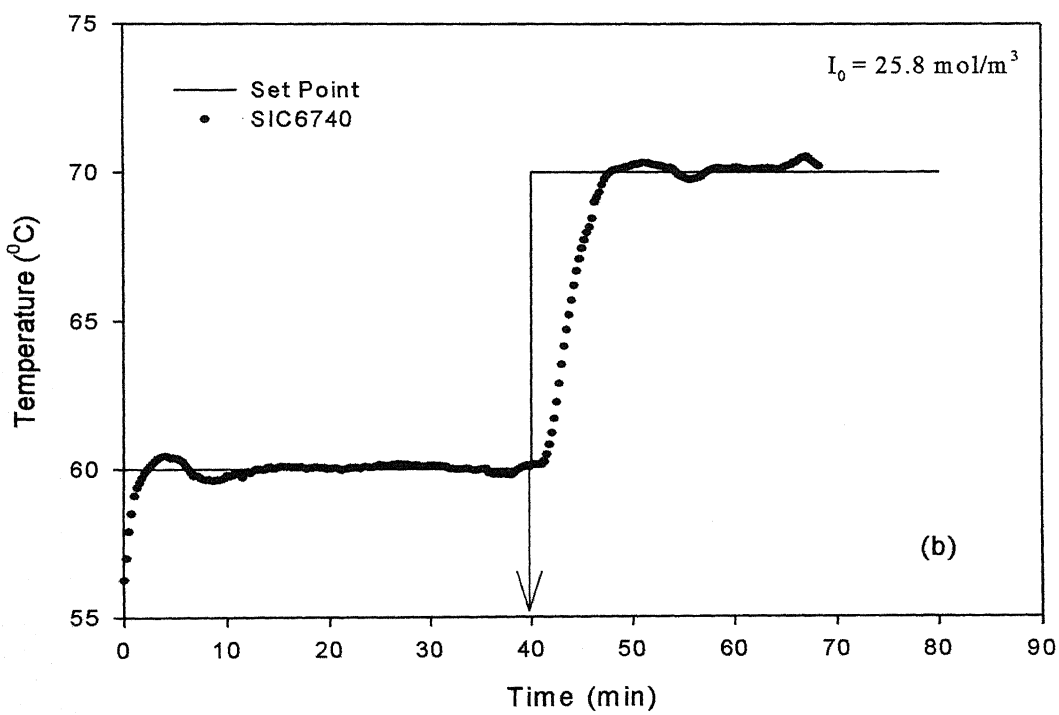


Figure 3.15.1 Temperature histories for step change from 60°C to 70°C at 40 min (a) replicate and (b) original.

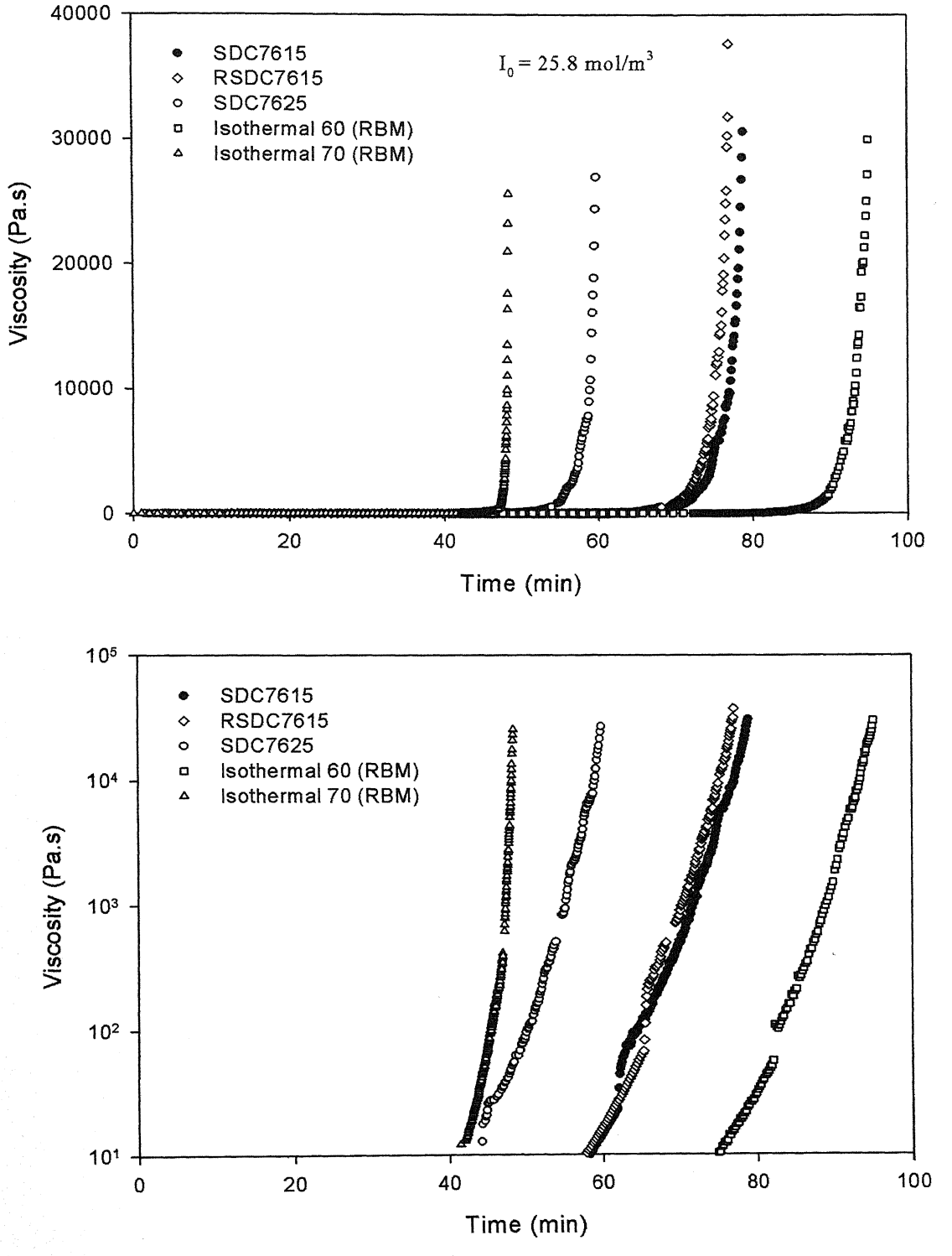


Figure 3.16 Experimental data on viscosity vs time for temperature step change from 70°C to 60°C at different times (15 min and 25 min). $I_0 = 25.8$ mol/m^3.

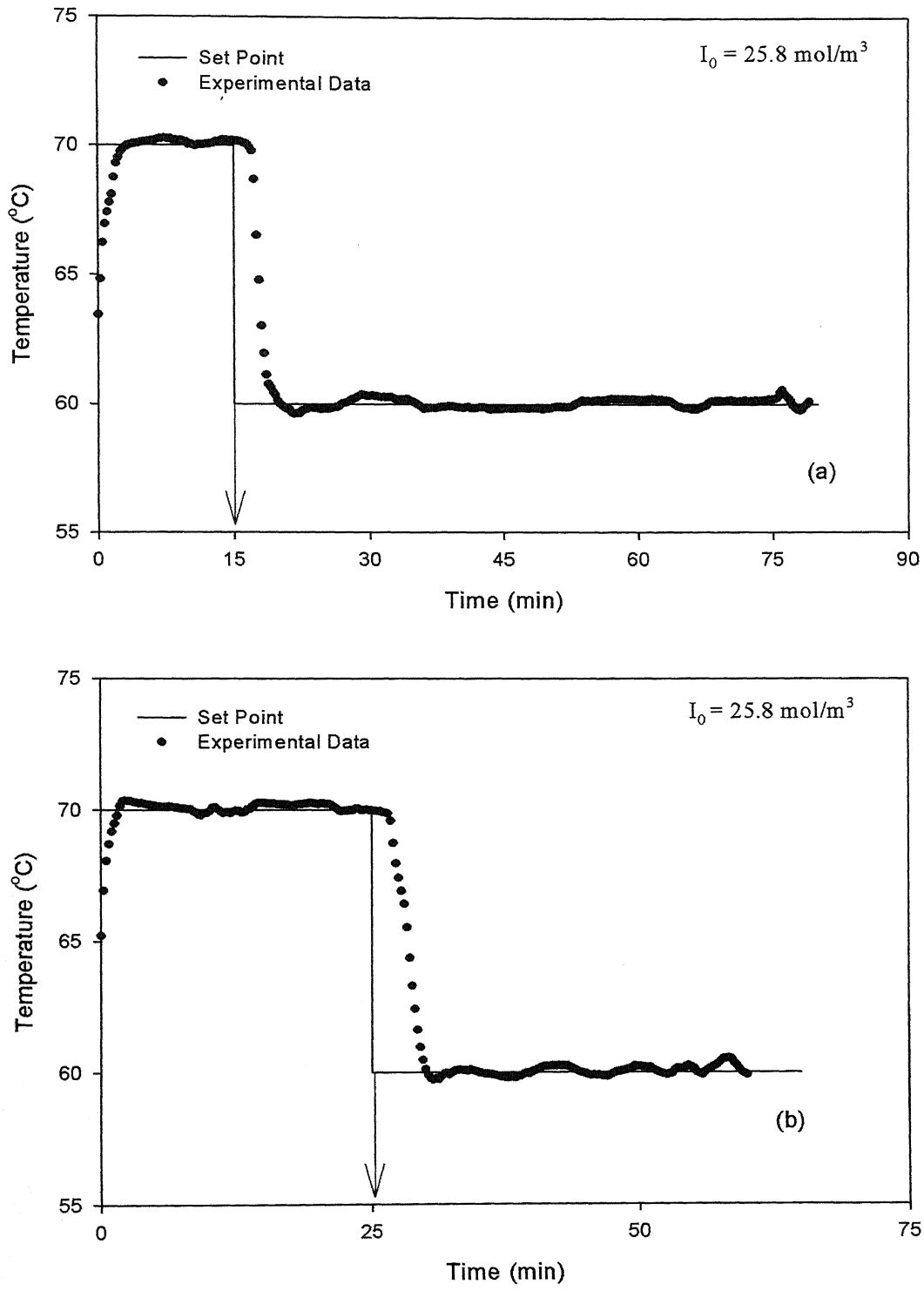
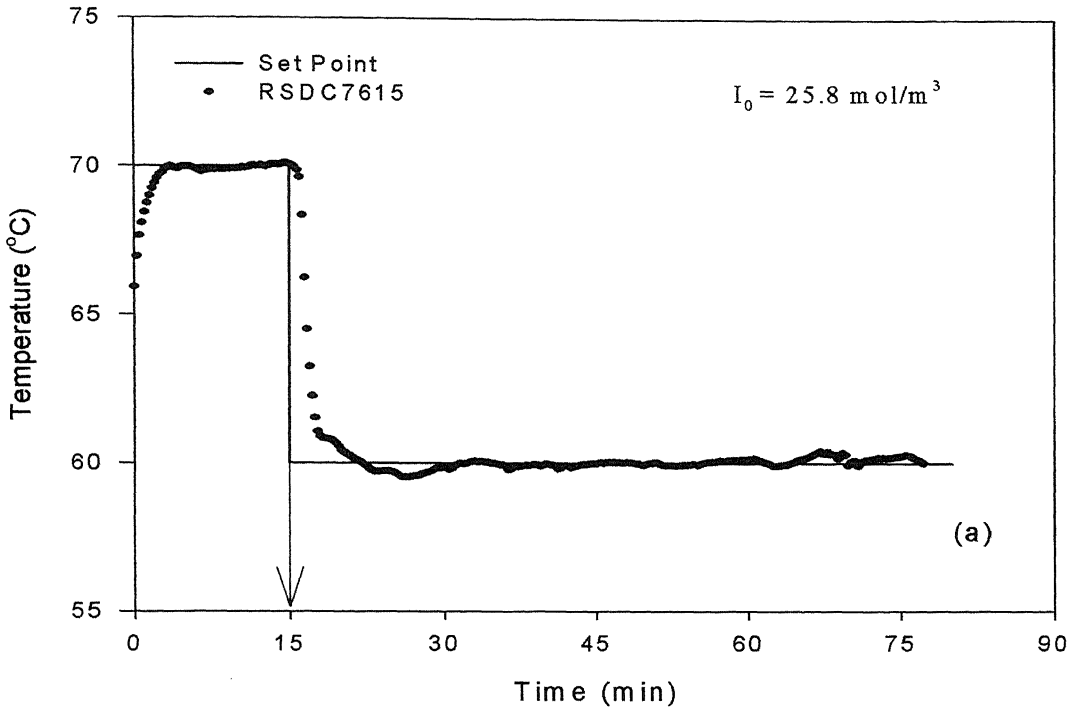


Figure 3.17 Temperature histories for step change from 70°C to 60°C at (a) 15 min and (b) 25 min.



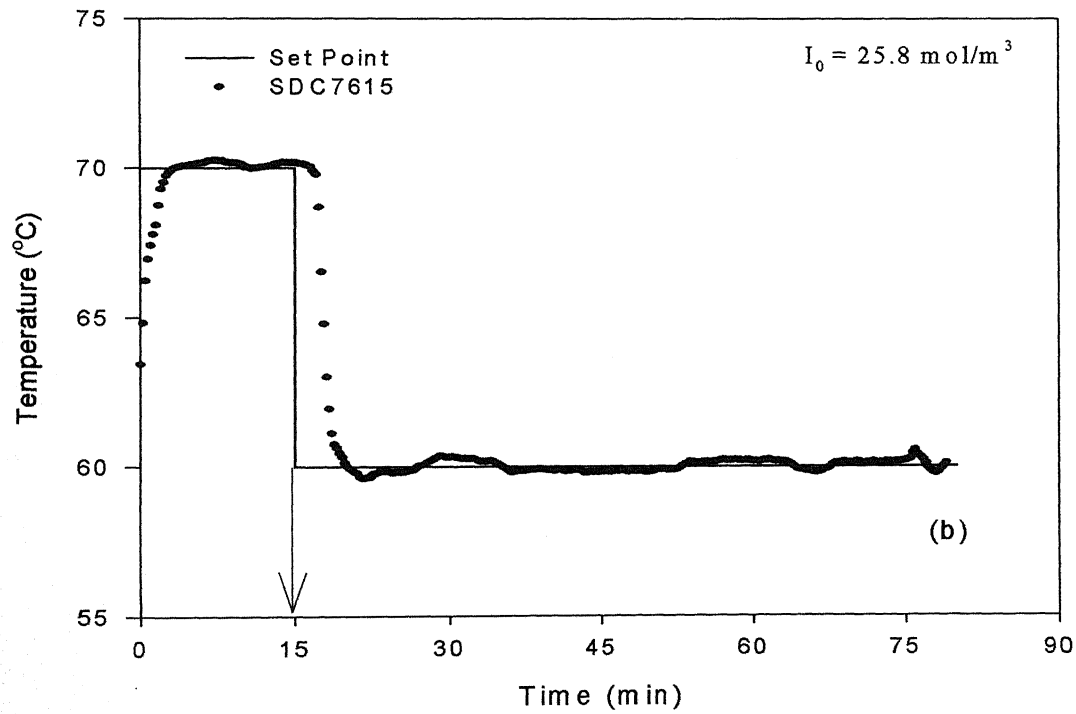


Figure 3.17.1 Temperature histories for step change from 70°C to 60°C at 15 min (a) replicate and (b) original.

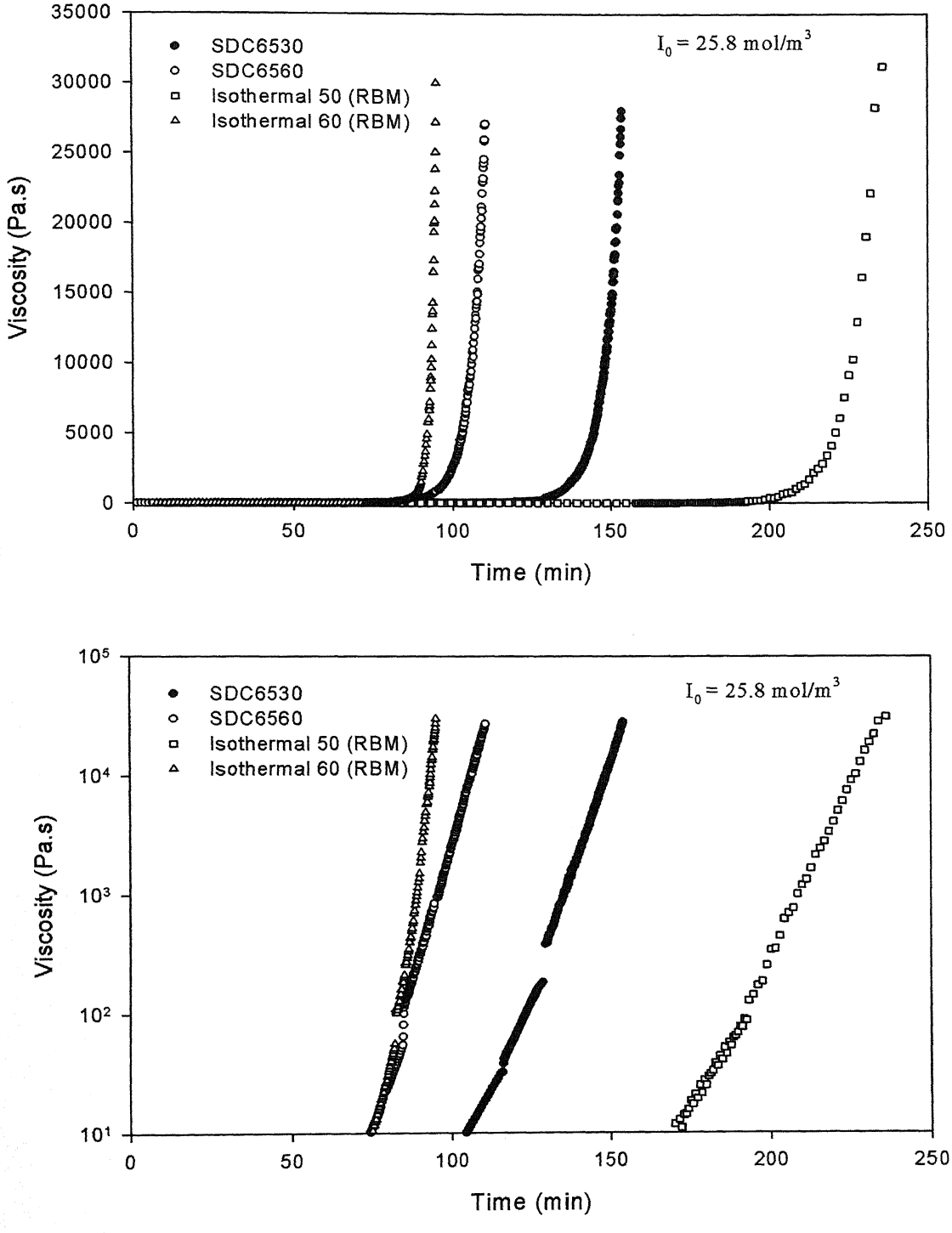


Figure 3.18 Experimental data on viscosity vs time for temperature step change from 60°C to 50°C at different times (30 min and 60 min). $I_0 = 25.8$ mol/m^3.

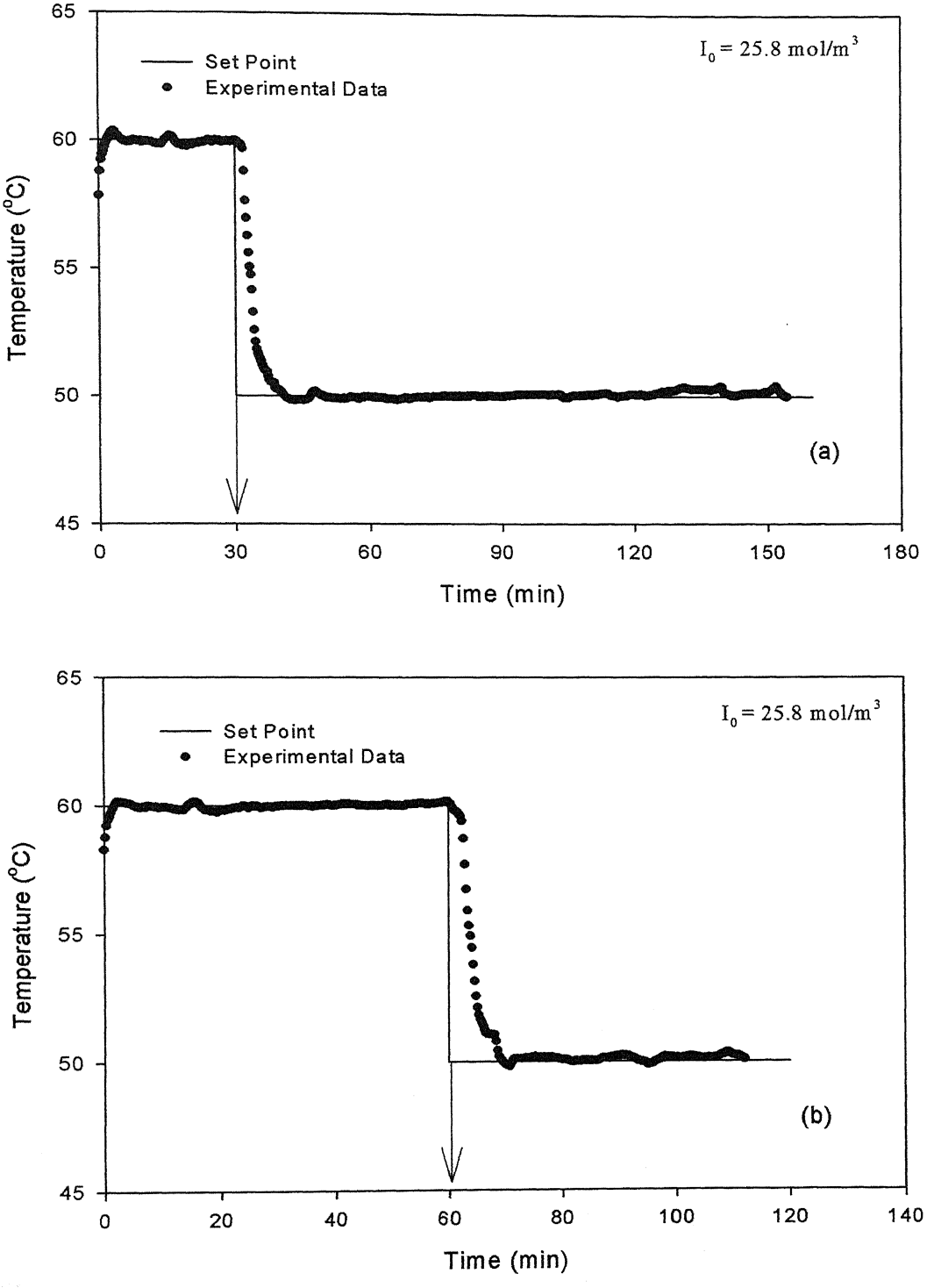


Figure 3.19 Temperature histories for step change from 60°C to 50°C at (a) 30 min and (b) 60 min.

APTER 4

NCLUSION AND SUGGESTION FOR FUTURE WORK

: polymerization of MMA was carried out in a rheometer-reactor assembly, which was ified by by Mankar (29). A series of experiments under different temperature histories, increase (50°C to 60°C and 60°C to 70°C) and step decrease (70°C to 60°C and 60°C to ?), have been conducted. The viscosity of the polymerizing mixture has been measured inuously and recorded in the PC. The temperature of the reaction mixture was measured the help of Pt100 sensor dipped into the reaction mixture. The viscosity vs. time data been plotted along with the isothermal data of Mankar (29). One set of data from each of temperature histories has been replicated to ensure the reproducibility of the data. The erated data are internally consistent and comparable to those reported in literature and ce should prove to be valuable in the development of better correlations for prediction of osity and its subsequent use in the development of soft sensors for reactor control.

temperature control in the annulus of the viscometer was achieved by circulating hot er from the Julabo bath, whose temperature is controlled with a PID type controller and its internal sensor. To achieve a satisfactory control of temperature of reaction mixture it necessary to manually adjust the set point of the bath. Better control of temperature can achieved if the Pt100 external sensor can be used to control the bath temperature.

re is considerable scope to expand this work to cover a wider range of data. More erimental data can be generated with different temperature histories, like sinusoidal perature history with different frequencies and amplitudes, double step increase, double

step decrease, which will increase the horizon of applicability of the software sensor. The range of the reaction temperature should be increased beyond 70°C to make the measurements more akin to industrial practice.

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APPENDIX A

List of Experimental Runs

	Sr. No.	Description Of the Run	Run No.
SET- A	1	Isothermal 70°C (I_0 = 15.48 mol/m^3)	IS7015
	2	Isothermal 60°C (I_0 = 25.8 mol/m^3)	IS6025
SET- B [I_0 = 15.48 mol/m^3]	**STEP INCREASE OF TEMPERATURE**		
	3	From 50°C to 60°C at 100 min	SIB56100
	4.	From 50°C to 60°C at 150 min	SIB56150
	5	From 60°C to 70°C at 40 min	SIB6740
	6	From 60°C to 70°C at 60 min	SIB6760
	7	Replicate Run of SIB56100	RSIB56100
	STEP DECREASE OF TEMPERATURE		
	8	From 70°C to 60°C at 15 min	SDB7615
	9	From 70°C to 60°C at 25 min	SDB7625
	10	From 60°C to 50°C at 30 min	SDB6530
	11	From 60°C to 50°C at 60 min	SDB6560
	12	Replicate Run of SDB6550	RSDB6560
SET- C [I_0 = 25.8 mol/m^3]	**STEP INCREASE OF TEMPERATURE**		
	13	From 50°C to 60°C at 100 min	SIC56100
	14	From 50°C to 60°C at 150 min	SIC56150
	15	From 60°C to 70°C at 40 min	SIC6740
	16	From 60°C to 70°C at 60 min	SIC6760
	17	Replicate Run of SIC6740	RSIC6740
	STEP DECREASE OF TEMPERATURE		
	18	From 70°C to 60°C at 15 min	SDC7615
	19	From 70°C to 60°C at 25 min	SDC7625
	20	From 60°C to 50°C at 30 min	SDC6530
	21	From 60°C to 50°C at 60 min	SDC6560
	22	Replicate Run of SDC7615	RSDC7615

APPENDIX B

Shear Rate Setting for Haake Software along with Actual Value of Shear Rate and Shear Stress

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Table B-1 Shear Rate Setting For IS7015, IS6025, SIB56100

Run No.	Time (min)	Shear Rate Setting (s^{-1})	Actual Shear Rate (s^{-1})	Shear Stress (Pa)
IS7015	18.0100	10.0000	10.0500	0.0000
	27.0100	10.0000	10.0500	0.0000
	36.0000	10.0000	10.0400	0.0000
	44.1200	10.0000	10.0400	6.8980
	48.0140	2.0000	2.0470	10.4000
	49.0190	2.0000	2.0490	13.5300
	51.0130	2.0000	2.0470	21.8000
	52.4210	0.1000	0.1472	3.5410
	55.0240	0.1000	0.1385	9.8500
	58.0140	0.1000	2.0470	334.5000
	61.0280	0.1000	0.1370	255.8000
	63.5330	0.1000	0.1385	3693.0000
IS6025	15.6000	10.0000	10.0400	0.0000
	29.9100	10.0000	10.0400	0.0000
	45.5100	10.0000	10.0400	0.0000
	55.0070	10.0000	10.0500	2.4260
	61.8100	10.0000	10.0300	17.6400
	62.1140	2.0000	2.5880	4.9620
	65.1130	2.0000	2.0260	6.2480
	69.1000	2.0000	2.0210	10.8400
	71.7000	2.0000	2.0180	16.4200
	72.2100	0.1000	0.1718	3.1240
	77.0200	0.1000	0.1247	4.9010
	82.1142	0.1000	0.1189	13.9100
	87.3940	0.1000	0.1348	63.4600
	92.0200	0.1000	0.1370	346.0000
	96.3000	0.1000	0.1334	3732.0000
SIB56100	44.4100	10.0000	10.0300	0.0000
	84.3100	10.0000	10.0300	0.0000
	104.1000	10.0000	10.0300	0.0000
	105.2000	2.0000	2.5800	0.0000
	115.3000	0.2000	0.2508	0.0000
	119.3000	0.2000	0.2095	0.0000
	127.0040	2.0000	2.0020	1.5680
	130.1020	2.0000	2.0050	2.0580
	130.7110	0.2000	1.9990	2.3650
	168.0200	0.2000	0.2182	14.4000
	184.0590	0.1500	0.1733	134.4000
	200.5960	0.1000	0.1256	2725.0000
	201.3297	0.1000	0.1277	3445.0000

Table B-2 Shear Rate Setting For SIB56150, SIB6740, SIB6760, RSIB56100

Run No.	Time (min)	Shear Rate Setting (s^{-1})	Actual Shear Rate (s^{-1})	Shear Stress (Pa)
SIB56150	40.8200	10.0000	9.9900	0.0000
	80.4200	10.0000	10.0000	0.0000
	120.0000	10.0000	10.0200	0.0000
	164.7900	10.0000	10.0000	19.3500
	174.6108	0.1000	0.1109	1.2130
	184.6100	0.1000	0.1087	3.7240
	194.8100	0.1000	0.1102	10.6500
	204.0100	0.1000	0.1109	34.5400
	214.1240	0.1000	0.1116	169.8000
	229.3200	0.1000	0.1203	3733.0000
SIB6740	20.7100	10.0000	10.0400	0.0000
	36.9100	10.0000	10.0400	0.0000
	44.1100	10.0000	10.0400	0.0000
	45.3100	2.0000	2.2210	0.0000
	49.2000	2.0000	2.0220	0.0000
	53.1100	2.0000	2.0200	0.0000
	56.1100	2.0000	2.0260	0.0000
	59.7100	2.0000	2.0200	0.0000
	62.0220	0.1000	0.1348	0.1348
	65.0220	0.1000	0.1399	0.8699
	68.3350	0.1000	0.1421	1.9720
	71.1200	0.1000	0.1385	2.4630
	74.1600	0.1000	0.1377	8.7720
	77.0480	0.1000	0.1406	41.1200
	80.9540	0.1000	0.1375	3746.0000
SIB6760	27.3000	10.0000	10.0500	0.0000
	52.0100	10.0000	10.0300	0.0000
	63.7000	10.0000	10.0300	0.0000
	65.4100	2.0000	2.0970	0.0000
	69.9100	2.0000	2.0240	0.0000
	75.0700	2.0000	2.0440	4.7900
	80.1090	2.0000	2.0450	12.6900
	84.7200	2.0000	2.0430	34.1900
	89.0440	0.1000	0.1399	11.7500
	91.0210	0.1000	0.1363	22.7800
	93.0110	0.1000	0.1406	47.4000
	95.0110	0.1000	0.1392	118.2000
	98.4060	0.1000	0.1450	3288.0000
RSIB56100	3.6090	10.0000	10.0100	0.0000
	44.4100	10.0000	10.0300	0.0000
	86.0000	10.0000	10.0300	0.0000
	123.7000	10.0000	10.0300	0.0000
	127.9120	2.0000	2.0130	0.7106
	138.5100	2.0000	2.0330	2.4870
	147.1100	2.0000	2.0370	6.2850
	147.3000	2.0000	2.0320	6.5300
	149.2063	0.1000	0.1298	0.7473
	159.2100	0.1000	0.1276	2.7080
	169.3100	0.1000	0.1327	8.5880
	180.4118	0.1000	0.1341	34.4500
	190.0000	0.1000	0.1341	243.6000
	199.1000	0.1000	0.1332	3679.0000

Table B-3 Shear Rate Setting For SDB7615, SDB7625, SDB6530, SDB6560

Run No.	Time (min)	Shear Rate Setting (s^{-1})	Actual Shear Rate (s^{-1})	Shear Stress (Pa)
SDB7615	13.5100	10.0000	10.0400	0.0000
	24.3100	10.0000	10.0500	0.0000
	35.1300	10.0000	10.0500	0.0000
	48.0100	2.0000	2.0260	0.7596
	52.2100	2.0000	2.0260	1.0660
	56.8000	2.0000	2.0270	2.2910
	58.1200	0.1000	0.1254	0.7596
	68.2300	0.1000	0.1327	1.8620
	78.7160	0.1000	0.1399	3.3570
	88.1100	0.1000	0.1414	17.5700
	98.0100	0.1000	0.1406	191.0000
	108.6000	0.1000	0.1330	3754.0000
SDB7625	22.8000	10.0000	10.0400	0.0000
	42.0100	10.0000	10.0300	0.0000
	52.8200	10.0000	10.0400	36.7100
	54.9050	2.0000	2.0390	15.6600
	55.7462	2.0000	2.0090	16.4500
	56.3887	2.0000	2.0420	18.2300
	57.0100	0.1000	0.1399	2.4260
	62.1250	0.1000	0.1298	5.8560
	67.3300	0.1000	0.1399	18.4800
	72.0130	0.1000	0.1414	71.1700
	77.1120	0.1000	0.1435	308.6000
	82.2070	0.1000	0.1363	1364.0000
	85.7070	0.1000	0.1330	3695.0000
SDB6530	44.2100	10.0000	10.0500	0.0000
	85.2500	10.0000	10.0400	0.0000
	117.9100	10.0000	10.0300	17.4700
	118.4120	2.0000	2.0830	3.8710
	130.0100	2.0000	2.0180	8.8330
	139.8130	2.0000	2.0340	14.0800
	142.9100	2.0000	2.0420	17.4500
	144.0050	0.1000	0.1312	2.1320
	159.1000	0.1000	0.1356	6.2360
	174.5160	0.1000	0.1305	16.3700
	190.7000	0.1000	0.1269	66.9700
	205.2080	0.1000	0.1269	218.2000
	220.9060	0.1000	0.1334	801.7000
	237.7000	0.1000	0.1275	3699.0000
SDB6560	33.6100	10.0000	9.9760	0.0000
	63.5100	10.0000	10.0000	0.0000
	93.6100	10.0000	9.9980	37.8900
	94.6100	2.0000	1.9820	9.5930
	100.0100	2.0000	1.9730	14.9800
	106.9000	2.0000	1.9760	22.8200
	113.4200	2.0000	1.9650	38.0800
	118.8000	2.0000	1.9670	60.0100
	120.1040	0.1000	0.0805	8.8580
	132.2100	0.1000	0.0783	27.7900
	144.0000	0.1000	0.0798	100.4000
	166.0100	0.1000	0.0885	1155.0000
	177.6100	0.1000	0.0914	3725.0000

Table B-4 Shear Rate Setting For RSDB6560, SIC56100, SIC56150, SIC6740

Run No.	Time (min)	Shear Rate Setting (s^{-1})	Actual Shear Rate (s^{-1})	Shear Stress (Pa)
RSDB6560	30.0100	10.0000	9.9900	0.0000
	60.0100	10.0000	10.0200	0.0000
	99.3700	10.0000	10.0400	65.0300
	106.0100	2.0000	2.0190	26.3200
	112.3100	2.0000	2.0190	42.3700
	118.0200	2.0000	2.0230	69.0100
	124.8000	2.0000	2.0230	127.3000
	130.0000	0.1000	0.1341	30.8600
	140.0000	0.1000	0.1348	95.0600
	150.0000	0.1000	0.1363	308.1000
	160.0100	0.1000	0.1312	890.5000
	170.0100	0.1000	0.1298	2250.0000
	175.2100	0.1000	0.1261	3701.0000
SIC56100	25.2100	10.0000	10.0300	0.0000
	50.4100	10.0000	10.0300	0.0000
	100.8000	10.0000	10.0300	5.0110
	119.1000	10.0000	10.0300	194.8000
	120.4285	2.0000	2.0230	53.7000
	121.5490	2.0000	2.0330	68.6500
	123.0810	2.0000	2.0370	92.1100
	124.9100	2.0000	2.0330	138.9000
	126.0108	0.1000	0.1290	17.4100
	128.0040	0.1000	0.1312	31.3100
	130.0150	0.1000	0.1254	66.6600
	132.0160	0.1000	0.1254	178.3000
	134.3070	0.1000	0.1305	3446.0000
SIC56150	40.6100	10.0000	10.0300	0.0000
	75.4000	10.0000	10.0300	0.0000
	110.2670	10.0000	10.0300	7.7800
	146.3000	10.0000	10.0300	76.5100
	148.0200	2.0000	2.0130	18.6800
	154.0100	2.0000	2.0130	25.9700
	161.2100	2.0000	2.0180	54.4000
	164.0090	0.1000	0.1356	8.6250
	171.2010	0.1000	0.1312	28.2300
	178.4200	0.1000	0.1377	116.3000
	183.2130	0.1000	0.1356	344.1000
	190.0080	0.1000	0.1341	3430.0000
	190.1070	0.1000	0.1305	3625.0000
SIC6740	20.7100	10.0000	10.0300	0.0000
	32.4100	10.0000	10.0300	0.0000
	43.2010	10.0000	10.0300	4.1900
	44.6100	2.0000	2.0170	0.9434
	47.4120	2.0000	2.0040	1.3600
	53.6080	2.0000	2.0310	5.4640
	56.6070	2.0000	2.0380	11.9600
	57.9100	0.1000	0.1247	1.6050
	59.1100	0.1000	0.1312	2.8300
	61.1000	0.1000	0.1261	8.4660
	63.0100	0.1000	0.1225	43.2600
	65.1100	0.1000	0.1327	866.3000
	67.4800	0.1000	0.1276	3574.0000

Table B-5 Shear Rate Setting For SIC6760, RSIC6740, SDC7615, SDC7625

Run No.	Time (min)	Shear Rate Setting (s^{-1})	Actual Shear Rate (s^{-1})	Shear Stress (Pa)
SIC6760	20.3000	10.0000	10.0300	0.0000
	35.0000	10.0000	10.0300	0.0000
	50.1750	10.0000	10.0500	4.5820
	65.6500	10.0000	10.0400	57.1400
	66.4100	2.0000	2.0920	14.2600
	68.0100	2.0000	2.0270	20.0800
	71.4100	2.0000	2.0210	54.2600
	72.9600	2.0000	2.0210	102.2000
	74.5200	0.1000	0.1334	39.6200
	75.5000	0.1000	0.1247	59.7800
	76.5100	0.1000	0.1232	176.1000
	77.5100	0.1000	0.1334	1262.0000
	78.8100	0.1000	0.1319	3656.0000
RSIC6740	10.0000	10.0000	9.8890	0.0000
	25.0000	10.0000	9.8020	0.0000
	38.1076	10.0000	10.0300	1.2880
	45.1430	10.0000	10.0300	3.5750
	46.0090	2.0000	2.0100	1.5830
	49.0050	2.0000	2.0310	1.2990
	53.2070	2.0000	2.0340	4.4840
	57.8040	2.0000	2.0320	13.0000
	59.0100	0.1000	0.1261	1.4210
	61.6100	0.1000	0.1261	5.8930
	63.0000	0.1000	0.1327	15.6300
	65.1100	0.1000	0.1276	276.9000
	68.3700	0.1000	0.1319	3449.0000
SDC7615	11.7200	10.0000	9.9980	0.0000
	29.7100	10.0000	10.0000	0.0000
	46.4100	10.0000	10.0100	10.6100
	47.1100	2.0000	1.9970	4.2390
	53.1100	2.0000	1.9880	7.3630
	56.1100	2.0000	1.9920	11.5900
	61.7160	2.0000	2.0080	45.4800
	62.2120	0.1000	0.1189	5.8440
	66.3150	0.1000	0.1066	17.4200
	70.4100	0.1000	0.1095	66.0600
	74.5000	0.1000	0.1102	398.9000
	78.8100	0.1000	0.1138	3487.0000
SDC7625	10.5100	10.0000	10.0000	0.0000
	18.2100	10.0000	10.0300	0.0000
	26.6100	10.0000	10.0200	0.0000
	34.3100	10.0000	10.0300	1.0410
	35.5600	2.0000	2.0160	1.1390
	39.8010	2.0000	2.0190	6.1750
	41.3500	2.0000	2.0240	8.1960
	43.5910	2.0000	2.0160	14.2000
	44.3130	0.1000	0.1653	2.9280
	47.5100	0.1000	0.1160	4.7660
	50.0100	0.1000	0.1138	10.8900
	53.4200	0.1000	0.1138	48.0800
	56.6090	0.1000	0.1196	301.9000
	59.8130	0.1000	0.1240	3345.0000

Table B-6 Shear Rate Setting For SDC6530, SDC6560, RSDC7625

Run No.	Time (min)	Shear Rate Setting (s^{-1})	Actual Shear Rate (s^{-1})	Shear Stress (Pa)
SDC6530	2.4060	10.0000	10.0100	0.0000
	30.2010	10.0000	10.0300	0.0000
	60.2100	10.0000	10.0300	0.6983
	90.1700	10.0000	10.0400	25.6400
	115.3300	10.0000	10.0400	315.5000
	116.4100	2.0000	2.0900	87.6400
	120.0000	2.0000	2.0220	134.5000
	124.2100	2.0000	2.0260	230.0000
	128.6100	2.0000	2.0270	373.8000
	130.0073	0.1000	0.1414	55.7000
	135.0070	0.1000	0.1385	128.0000
	140.0100	0.1000	0.1406	308.7000
	145.1100	0.1000	0.1414	768.9000
	150.2100	0.1000	0.1421	1953.0000
	153.8100	0.1000	0.1341	3763.0000
SDC6560	1.2030	10.0000	10.0300	0.0000
	15.6000	10.0000	10.0200	0.0000
	36.0000	10.0000	10.0300	0.0000
	47.6630	10.0000	10.0300	2.3400
	68.9600	10.0000	10.0300	30.5800
	70.1100	2.0000	2.0230	8.3430
	73.1100	2.0000	2.0130	15.8200
	76.1100	2.0000	2.0100	26.7200
	79.7200	2.0000	2.0080	48.9600
	84.2100	2.0000	2.0140	104.6000
	85.0100	0.1000	0.1225	14.7100
	91.1100	0.1000	0.1174	48.1000
	98.5120	0.1000	0.1305	248.7000
	104.6160	0.1000	0.1392	1001.0000
	110.7100	0.1000	0.1334	3617.0000
RSDC7625	3.7130	10.0000	9.9540	0.0000
	14.9000	10.0000	9.9610	0.0000
	30.1100	10.0000	9.9760	0.0000
	44.4020	10.0000	9.9830	8.5270
	54.9000	10.0000	9.9900	51.7800
	55.6100	2.0000	2.0400	13.5500
	57.0100	2.0000	1.9730	17.1000
	60.0100	2.0000	1.9730	33.2100
	62.0100	2.0000	1.9730	53.9800
	65.0000	2.0000	1.9740	126.1000
	65.5100	0.1000	0.1218	19.1900
	68.0000	0.1000	0.0761	37.0100
	71.0280	0.1000	0.0885	124.5000
	73.0220	0.1000	0.0921	329.4000
	76.9560	0.1000	0.0964	3632.0000

APPENDIX C

Experimental Data on Viscosity vs. Time

Table C-1 Data For Run IS7015

Time (min)	Viscosity (Pa.s)	Temperature (°C)
44.1200	0.6869	69.8800
46.1242	2.5410	70.0800
47.1243	4.3040	70.0800
48.1130	5.1650	70.1200
49.1180	6.6250	70.2000
50.1230	8.4430	70.1400
51.1120	11.0300	70.1600
52.1240	18.0500	70.2900
53.1130	29.5100	70.2200
54.1180	45.6100	69.8100
54.6120	59.7100	69.9400
55.2220	72.8300	70.2200
55.7160	85.7300	70.3300
56.2110	98.9100	70.2800
56.5240	113.3000	70.1700
56.7210	118.5000	70.1500
56.9100	125.2000	70.1700
57.6185	133.7000	70.2500
57.8163	146.9000	70.2600
58.0140	163.4000	70.2600
58.2120	175.2000	70.3800
58.4260	186.8000	70.3700
58.6240	199.6000	70.2400
58.8210	213.7000	70.0400
58.9353	340.8000	69.6000
59.0342	460.8000	69.5200
59.1330	583.9000	69.5200
59.3307	667.1000	69.6800
59.4297	726.0000	69.9000
59.6273	894.7000	60.1600
59.7262	944.1000	70.2400
60.2200	1104.0000	70.3500
60.4350	1267.0000	70.3000
60.6330	1369.0000	70.2800
60.7310	1530.0000	70.2600
60.9290	1689.0000	70.1300
61.0280	1867.0000	70.0400
61.1270	1929.0000	69.9600
61.2260	2103.0000	69.8800
61.3250	2252.0000	69.8200
61.4230	2352.0000	69.7900
61.5220	2575.0000	69.7400
61.6210	2825.0000	69.6800
61.9340	3467.0000	70.1000
62.1320	4333.0000	70.3200
62.3300	5025.0000	70.3000
62.4280	5552.0000	70.3000

62.5270	6316.0000	70.2300
62.6260	7252.0000	70.2200
62.7250	8209.0000	70.2000
62.8240	9505.0000	70.1700
62.9230	11110.0000	70.1300
63.0220	12450.0000	70.1100
63.1210	14730.0000	70.2200
63.2360	18050.0000	70.3400
63.3350	20890.0000	70.3600
63.4340	24460.0000	70.3500
63.5330	26660.0000	70.3000

Table C-2 Data For Run IS6025

Time (min)	Viscosity (Pa.s)	Temperature (°C)
48.4160	0.1990	59.9000
54.4140	0.2173	59.9000
56.8030	0.4191	59.8600
58.8110	1.1780	59.9700
60.9040	1.5450	59.7800
62.9050	2.3670	60.0000
64.9150	3.0930	59.9100
66.9100	4.0350	59.7200
69.1000	5.3660	59.7300
71.1100	7.5350	59.9900
72.6100	18.9400	60.0400
75.1100	25.9600	60.2000
76.8100	37.4400	59.8400
78.3000	47.6500	59.5900
79.8000	62.6300	59.7400
81.4100	87.4600	59.7900
83.0040	134.5000	59.9200
84.5040	203.4000	60.0300
86.0030	317.2000	60.1200
87.5100	477.3000	60.1200
88.0200	574.9000	60.1400
88.5150	682.3000	60.1400
88.9820	788.2000	60.1600
89.2630	887.9000	60.1630
89.6250	970.8000	60.1200
90.0200	1159.0000	60.1300
90.5150	1422.0000	60.1330
91.0650	1732.0000	60.1200
91.5100	1986.0000	60.1300
91.7900	2212.0000	60.1100
91.8230	2382.0000	60.1200
92.0700	2603.0000	60.1300
92.2700	2712.0000	60.1300
92.4700	2931.0000	60.1200
92.9000	3417.0000	60.1000
93.5153	4024.0000	60.1700
94.0097	5362.0000	60.2000
94.2075	6033.0000	60.2000
94.5040	7200.0000	60.2200
94.8010	8267.0000	60.2500
94.9160	8181.0000	60.2600
95.0150	8661.0000	60.2700
95.1140	8662.0000	60.2700
95.2130	9350.0000	60.2700
95.3110	9812.0000	60.2500

95.4100	10190.0000	60.2400
95.5090	11130.0000	60.2100
95.6080	12010.0000	60.1800
95.7070	13020.0000	60.1400
95.8060	15170.0000	60.1200
95.9050	17290.0000	60.0900
96.0030	20050.0000	60.0700
96.1020	22390.0000	60.0200
96.2010	25020.0000	59.9300
96.3000	27970.0000	59.9500

Table C-3 Data For Run SIB56100

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
1.2030	0.0000	49.5200	50.0000
21.6000	0.0000	50.0300	50.0000
32.4100	0.0000	50.0000	50.0000
52.8100	0.0000	50.0000	50.0000
73.5100	0.0000	50.0700	50.0000
94.2100	0.0000	50.0900	50.0000
104.1000	0.0000	58.3400	60.0000
115.0000	0.0000	60.1200	60.0000
122.1030	0.4255	60.1900	60.0000
124.0140	0.5298	59.8500	60.0000
126.1140	0.7047	59.6800	60.0000
128.1080	0.9358	59.9100	60.0000
130.5140	1.1800	59.8700	60.0000
132.8100	4.1120	59.8700	60.0000
135.3200	5.5390	59.8900	60.0000
138.1100	6.2080	59.9500	60.0000
140.3200	7.8520	59.9400	60.0000
142.6120	8.2570	59.9400	60.0000
145.0170	9.4810	59.9500	60.0000
147.0190	10.6300	59.9100	60.0000
149.2100	12.3900	59.9500	60.0000
151.9200	14.2200	60.0200	60.0000
154.2100	16.0500	60.0200	60.0000
156.3100	19.9000	60.0200	60.0000
158.5200	22.9900	60.0500	60.0000
160.7300	28.3500	60.0700	60.0000
162.8300	35.8900	60.0400	60.0000
164.9200	46.2400	60.3300	60.0000
166.7200	59.3900	60.1300	60.0000
168.9100	66.9400	59.9500	60.0000
171.1200	84.7700	59.9700	60.0000
172.2575	97.2000	59.9500	60.0000
173.4600	119.7000	59.9300	60.0000
174.8610	146.7000	59.9500	60.0000
176.1630	177.2000	60.0100	60.0000
177.4607	236.4000	60.0300	60.0000
179.3560	320.5000	60.0800	60.0000
180.9540	411.9000	60.1200	60.0000
182.2630	569.7000	60.1300	60.0000
183.7630	706.1000	60.1600	60.0000
185.1630	933.1000	60.1700	60.0000
186.3660	1182.0000	60.2100	60.0000
187.8600	1568.0000	60.1600	60.0000
189.2600	1992.0000	60.1700	60.0000
190.5600	2580.0000	60.1600	60.0000

192.0700	3530.0000	60.1800	60.0000
193.8600	5060.0000	60.2300	60.0000
195.1600	6974.0000	60.2000	60.0000
196.4600	8854.0000	60.2500	60.0000
197.3600	10930.0000	60.3300	60.0000
199.0920	14890.0000	60.3200	60.0000
199.1910	15210.0000	60.3100	60.0000
199.9980	17680.0000	60.2600	60.0000
200.3960	19950.0000	60.2000	60.0000
200.8353	20080.0000	60.1700	60.0000
201.0330	22910.0000	60.1000	60.0000
201.1320	24120.0000	60.0900	60.0000
201.3297	26980.0000	60.0700	60.0000

Table C-4 Data For Run SIB56150

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
4.8120	0.0000	50.0600	50.0000
30.0100	0.0000	50.0100	50.0000
55.2000	0.0000	49.9500	50.0000
78.0100	0.0000	50.0300	50.0000
102.0000	0.0000	50.0600	50.0000
120.0000	0.0000	50.0400	50.0000
148.9500	0.7997	49.9600	50.0000
156.2000	0.9601	59.5500	60.0000
160.1700	1.1930	60.0800	60.0000
164.7900	1.9340	60.0700	60.0000
169.5100	2.9400	60.1400	60.0000
171.7000	3.6560	60.1200	60.0000
173.1000	4.1880	60.1200	60.0000
175.2040	10.9600	60.1900	60.0000
177.1150	15.2400	60.2500	60.0000
179.7100	18.6000	60.2800	60.0000
181.9010	23.3300	60.3100	60.0000
183.2030	29.5700	60.2700	60.0000
185.5000	37.9400	60.1700	60.0000
187.4100	42.1400	60.0000	60.0000
190.1100	61.8900	69.9700	60.0000
193.0100	81.6000	60.0600	60.0000
195.8100	107.2000	60.0500	60.0000
197.1200	129.9000	60.1100	60.0000
198.7100	157.7000	60.1300	60.0000
200.2000	182.1000	60.1400	60.0000
201.7000	234.7000	60.2300	60.0000
203.3000	281.5000	60.0600	60.0000
204.9220	338.8000	59.8300	60.0000
206.3230	413.2000	59.8500	60.0000
208.0200	531.3000	59.8800	60.0000
210.3170	809.2000	59.9200	60.0000
212.3110	1121.0000	59.9600	60.0000
214.1240	1521.0000	59.9400	60.0000
215.5100	1956.0000	59.9700	60.0000
217.1100	2576.0000	59.9200	60.0000
218.9300	3630.0000	59.9600	60.0000
220.3200	4792.0000	59.9800	60.0000
221.4200	5965.0000	59.9700	60.0000
222.2100	6986.0000	59.9800	60.0000
222.9200	7919.0000	59.9800	60.0000

223.3200	8826.0000	59.9700	60.0000
224.0200	9678.0000	60.0300	60.0000
224.4200	10970.0000	60.0500	60.0000
225.4200	12950.0000	60.1500	60.0000
225.6100	13700.0000	60.1400	60.0000
225.8100	14630.0000	60.2000	60.0000
226.2200	15720.0000	60.2500	60.0000
226.5200	16850.0000	60.2200	60.0000
226.6200	17380.0000	60.2000	60.0000
227.0100	18200.0000	60.1500	60.0000
227.1100	19640.0000	60.1300	60.0000
227.5300	20720.0000	60.1000	60.0000
227.7200	21670.0000	60.0700	60.0000
227.9200	22770.0000	60.0600	60.0000
228.0200	23510.0000	60.0600	60.0000
228.3200	25640.0000	60.0700	60.0000
228.5100	26980.0000	60.0600	60.0000
228.7100	27450.0000	60.0400	60.0000
229.0200	29190.0000	60.0200	60.0000
229.2200	30130.0000	60.0100	60.0000
229.3200	31020.0000	60.0000	60.0000

Table C-5 Data For Run SIB6740

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.7020	0.0000	60.3900	60.0000
13.5100	0.0000	59.9800	60.0000
27.0000	0.0000	59.9700	60.0000
36.0200	0.0000	60.0400	60.0000
45.0000	0.0000	69.7700	70.0000
61.7250	0.5423	70.0400	70.0000
62.6810	1.4460	70.1600	70.0000
63.7020	2.3750	70.1700	70.0000
64.6750	5.4540	70.1900	70.0000
65.5650	6.1220	70.1400	70.0000
66.5210	8.0090	70.0700	70.0000
67.7250	10.2600	70.0100	70.0000
69.0430	11.9100	69.9100	70.0000
70.0500	12.8800	69.9400	70.0000
71.1200	17.7800	70.0800	70.0000
72.2100	28.1000	70.2600	70.0000
73.5700	44.3500	70.2200	70.0000
74.5600	71.1000	70.2100	70.0000
75.5300	118.8000	70.1700	70.0000
76.4543	196.5000	70.1500	70.0000
77.0480	292.3000	70.1600	70.0000
77.5910	387.0000	70.1600	70.0000
77.9540	454.9000	70.1600	70.0000
78.3160	585.6000	70.1500	70.0000
78.4810	651.7000	70.1500	70.0000
78.6620	801.1000	70.1400	70.0000
78.7280	912.3000	70.1100	70.0000
78.7940	1012.0000	70.0900	70.0000
78.9100	1251.0000	70.0700	70.0000
79.0260	1384.0000	70.0700	70.0000
79.0920	1548.0000	70.0700	70.0000
79.1410	1639.0000	70.0600	70.0000
79.2070	1752.0000	70.0400	70.0000
79.2730	1827.0000	70.0400	70.0000
79.3230	1928.0000	70.0400	70.0000
79.3880	2062.0000	70.0300	70.0000

79.4540	2242.0000	70.0300	70.0000
79.5040	2385.0000	70.0200	70.0000
79.5700	2664.0000	70.0200	70.0000
79.6350	2826.0000	70.0200	70.0000
79.6850	2983.0000	70.0100	70.0000
79.7510	3220.0000	70.0000	70.0000
79.8000	3575.0000	70.0000	70.0000
79.8660	3888.0000	70.0000	70.0000
79.9320	4482.0000	70.0000	70.0000
79.9820	4614.0000	70.0000	70.0000
80.0480	5442.0000	70.0000	70.0000
80.1130	6116.0000	70.0100	70.0000
80.1630	6819.0000	70.0100	70.0000
80.2290	7484.0000	70.0100	70.0000
80.2950	8717.0000	70.0100	70.0000
80.3440	9525.0000	70.0100	70.0000
80.4100	10950.0000	70.0100	70.0000
80.4760	12820.0000	70.0100	70.0000
80.5250	13560.0000	70.0200	70.0000
80.5910	15820.0000	70.0200	70.0000
80.6410	17270.0000	70.0200	70.0000
80.7070	19440.0000	70.0300	70.0000
80.7730	21760.0000	70.0200	70.0000
80.8220	23850.0000	70.0200	70.0000
80.8880	25660.0000	70.0100	70.0000
80.9540	27240.0000	70.0100	70.0000

Table C-6 Data For Run SIB6760

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.6040	0.0000	60.0100	60.0000
13.0000	0.0000	59.8800	60.0000
24.7000	0.0000	60.0400	60.0000
39.0000	0.0000	60.0400	60.0000
52.0100	0.0000	60.1000	60.0000
65.0000	0.0000	70.9300	70.0000
70.0000	0.0000	69.4400	70.0000
71.2802	1.5010	69.5400	70.0000
73.8010	2.0410	69.7300	70.0000
76.3220	3.1520	70.0500	70.0000
78.8400	4.7680	70.2200	70.0000
80.9500	7.8260	69.9600	70.0000
82.0700	9.3580	69.8500	70.0000
83.0400	11.5000	69.9300	70.0000
84.0300	14.2300	69.9600	70.0000
85.6318	37.1800	69.9500	70.0000
87.0160	50.7800	70.0400	70.0000
88.0210	66.8800	70.1300	70.0000
89.0440	83.9700	70.2200	70.0000
90.0160	117.7000	70.2900	70.0000
91.0210	167.1000	70.4400	70.0000
92.0110	233.2000	70.5400	70.0000
93.0270	345.5000	70.5900	70.0000
94.0270	509.9000	70.7000	70.0000
95.0270	843.1000	70.8400	70.0000
95.1760	915.5000	70.8700	70.0000
95.3240	1002.0000	70.9600	70.0000
95.4720	1139.0000	71.0000	70.0000
96.1318	1426.0000	71.4100	70.0000
96.3132	1509.0000	71.8900	70.0000
96.7250	1893.0000	70.9600	70.0000

97.0220	2256.0000	70.5000	70.0000
97.3350	2741.0000	70.2000	70.0000
97.3840	2945.0000	70.1700	70.0000
97.4500	2998.0000	70.1300	70.0000
97.5160	3231.0000	70.1000	70.0000
97.5650	3434.0000	70.0300	70.0000
97.6310	3524.0000	69.9500	70.0000
97.6810	4119.0000	69.9200	70.0000
97.7470	4394.0000	69.8000	70.0000
97.8130	5064.0000	69.7600	70.0000
97.8620	5863.0000	69.7200	70.0000
97.9280	7177.0000	69.7000	70.0000
97.9940	8268.0000	69.6600	70.0000
98.0430	9632.0000	69.6500	70.0000
98.1090	11130.0000	69.6200	70.0000
98.1750	13110.0000	69.6100	70.0000
98.2240	15220.0000	69.5800	70.0000
98.2900	18220.0000	69.5700	70.0000
98.3560	21630.0000	69.5400	70.0000
98.4060	22670.0000	69.5100	70.0000

Table C-7 Data for Run RSBI56100

Time (Min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.4060	0.0000	50.1600	50.0000
27.6000	0.0000	50.0400	50.0000
43.2000	0.0000	49.9700	50.0000
70.4100	0.0000	49.9900	50.0000
99.0000	0.0000	50.0800	50.0000
105.5000	0.0000	60.5800	60.0000
118.5000	0.0000	60.0000	60.0000
127.9120	0.3531	60.0800	60.0000
130.9110	0.6203	60.0900	60.0000
133.7120	0.8606	59.9000	60.0000
137.9100	1.1650	59.9800	60.0000
140.9100	1.6750	59.9200	60.0000
144.1100	2.2190	59.9000	60.0000
147.3000	3.2130	59.8300	60.0000
150.9030	6.7790	59.9900	60.0000
154.1070	10.3600	60.1100	60.0000
157.6010	15.2000	60.0700	60.0000
160.3100	23.9000	60.0700	60.0000
162.9100	31.9000	60.1000	60.0000
165.7100	39.3100	60.0000	60.0000
167.1100	49.8500	60.0000	60.0000
169.6000	66.0200	60.0100	60.0000
172.2100	99.0400	60.0400	60.0000
174.6000	139.1000	60.0700	60.0000
177.0100	209.5000	60.1200	60.0000
180.4118	256.9000	60.2000	60.0000
182.6030	390.3000	60.2300	60.0000
184.5150	597.8000	60.3300	60.0000
185.8070	777.6000	60.1700	60.0000
187.0100	976.0000	60.1600	60.0000
188.1140	1240.0000	60.1100	60.0000
189.3010	1610.0000	60.1300	60.0000
190.6100	2083.0000	60.1500	60.0000
192.1100	2928.0000	60.1500	60.0000
193.4100	3966.0000	60.0800	60.0000

194.5100	5108.0000	59.8500	60.0000
195.7100	6697.0000	59.7600	60.0000
197.0100	9717.0000	59.6900	60.0000
197.2100	10020.0000	59.7200	60.0000
197.5000	10360.0000	59.7500	60.0000
197.6000	10440.0000	59.7900	60.0000
197.8000	10710.0000	59.7900	60.0000
198.0200	10740.0000	59.8100	60.0000
198.2100	10990.0000	59.7900	60.0000
198.3100	11470.0000	59.7700	60.0000
198.4100	12260.0000	59.7700	60.0000
198.5100	13590.0000	59.7600	60.0000
198.6100	15060.0000	59.7600	60.0000
198.7100	16930.0000	59.7500	60.0000
198.8100	19810.0000	59.7600	60.0000
198.9100	22810.0000	59.7600	60.0000
199.0000	25590.0000	59.7700	60.0000
199.1000	27620.0000	59.7700	60.0000

Table C-8 Data For Run SDB7615

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.7020	0.0000	70.2900	70.0000
9.0130	0.0000	69.9700	70.0000
14.4000	0.0000	70.0800	70.0000
18.0100	0.0000	67.8200	60.0000
28.0810	0.0000	60.2200	60.0000
39.0900	0.1181	59.9300	60.0000
43.4900	0.2036	60.0200	60.0000
48.0100	0.3750	59.6900	60.0000
50.4100	0.5570	59.7900	60.0000
52.8000	0.7074	60.0000	60.0000
55.0100	0.8878	60.1600	60.0000
57.1600	3.0000	59.8300	60.0000
59.4100	7.8290	59.7400	60.0000
61.6500	9.4710	59.8700	60.0000
63.0800	11.1100	59.9600	60.0000
65.0000	13.5400	60.0500	60.0000
67.2400	14.3500	60.1200	60.0000
69.2000	16.5300	60.0300	60.0000
71.3100	18.6500	59.9800	60.0000
74.0130	20.4900	60.2100	60.0000
76.0060	28.6700	60.1100	60.0000
78.8150	31.4900	59.8300	60.0000
81.2040	35.0100	59.9800	60.0000
83.2100	47.4900	60.1000	60.0000
85.2200	71.8400	60.1000	60.0000
87.2000	102.4000	60.1100	60.0000
89.2100	152.5000	60.1100	60.0000
91.2000	253.8000	60.0500	60.0000
93.2073	382.2000	60.0500	60.0000
93.3062	402.0000	60.0500	60.0000
95.0030	615.9000	60.2000	60.0000
96.0080	802.3000	60.2800	60.0000
97.0140	1059.0000	60.2900	60.0000
98.0100	1358.0000	59.7800	60.0000
99.0150	1749.0000	60.0200	60.0000
100.0030	2296.0000	60.1700	60.0000
101.1080	3041.0000	60.1400	60.0000
102.1130	4065.0000	60.1400	60.0000

103.1100	5269.0000	60.2000	60.0000
104.1100	6808.0000	59.8500	60.0000
104.2100	7277.0000	59.8600	60.0000
105.2000	9529.0000	60.1000	60.0000
106.0100	12030.0000	60.2400	60.0000
106.5000	14060.0000	60.2100	60.0000
107.0100	16310.0000	60.1700	60.0000
107.3100	17980.0000	60.1300	60.0000
107.5200	18300.0000	60.1000	60.0000
107.7100	19720.0000	60.2000	60.0000
107.8100	20520.0000	59.8300	60.0000
107.9100	21920.0000	59.8200	60.0000
108.0100	22510.0000	59.8200	60.0000
108.1100	23220.0000	59.8300	60.0000
108.2100	24040.0000	59.8300	60.0000
108.3100	24920.0000	59.8500	60.0000
108.4100	25860.0000	59.8600	60.0000
108.5000	26860.0000	59.8800	60.0000
108.6000	28230.0000	59.9000	60.0000

Table C-9 Data For Run SDB7625

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
1.2030	0.0000	68.6500	70.0000
12.0100	0.0000	69.9900	70.0000
24.0100	0.0000	69.8100	70.0000
30.0100	0.0000	66.5100	60.0000
44.4100	0.0927	59.6300	60.0000
45.6100	0.3609	59.8700	60.0000
46.8000	0.6357	60.0100	60.0000
48.0000	1.0990	59.9400	60.0000
49.2100	1.5570	59.9200	60.0000
50.4100	2.1730	59.9600	60.0000
51.6100	2.8660	59.9900	60.0000
52.8200	3.6560	59.9800	60.0000
54.0155	6.9780	59.5600	60.0000
55.4000	7.7440	59.6300	60.0000
56.0427	8.5660	59.6800	60.0000
57.0100	17.3400	59.5800	60.0000
58.5090	23.0300	59.6000	60.0000
59.3000	39.2300	59.9200	60.0000
62.1250	45.1300	59.9200	60.0000
63.4930	53.4000	59.9100	60.0000
64.6950	70.7600	69.9700	60.0000
65.6500	88.3200	60.0200	60.0000
66.6000	109.8000	60.0900	60.0000
67.5800	140.6000	60.1400	60.0000
68.3500	171.4000	60.2000	60.0000
69.6073	268.7000	60.2700	60.0000
70.5140	343.3000	60.3800	60.0000
71.5020	452.3000	60.3500	60.0000
72.5070	592.2000	60.3200	60.0000
73.5130	794.9000	60.3000	60.0000
74.3110	983.9000	60.2700	60.0000
75.1020	1268.0000	60.2900	60.0000
75.6130	1448.0000	60.2800	60.0000
76.0080	1624.0000	60.3000	60.0000
76.4030	1750.0000	60.3000	60.0000
77.1120	2150.0000	60.2700	60.0000
77.2110	2344.0000	60.2600	60.0000

77.7050	2701.0000	60.1800	60.0000
78.2160	3108.0000	60.0400	60.0000
78.7100	3635.0000	60.0800	60.0000
79.2100	4138.0000	60.1100	60.0000
80.3118	4919.0000	60.1300	60.0000
81.0040	6510.0000	60.1900	60.0000
81.5150	7561.0000	60.2300	60.0000
82.0090	9115.0000	60.2600	60.0000
82.6020	11480.0000	60.2500	60.0000
83.2120	12780.0000	60.3400	60.0000
83.4100	13530.0000	60.4000	60.0000
83.7060	14980.0000	60.4600	60.0000
83.9040	16520.0000	60.4800	60.0000
84.1020	17980.0000	60.4300	60.0000
84.3160	19540.0000	60.2300	60.0000
84.5140	20000.0000	59.9200	60.0000
84.8100	23340.0000	59.8800	60.0000
85.2130	25300.0000	59.9000	60.0000
85.4110	26160.0000	59.9300	60.0000
85.5100	26440.0000	59.9500	60.0000
85.6080	26910.0000	59.9600	60.0000
85.7070	27810.0000	59.9900	60.0000

Table C-10 Data For Run SDB6530

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
3.9050	0.0000	60.2100	60.0000
13.0000	0.0000	60.0000	60.0000
26.0000	0.0000	60.1530	60.0000
29.9100	0.0000	60.0400	50.0000
65.0000	0.0000	49.8600	50.0000
85.2500	0.0000	49.9400	50.0000
105.4000	0.3968	49.9800	50.0000
111.2000	0.8263	49.9500	50.0000
114.0050	1.3870	49.9800	50.0000
117.2020	1.7060	49.9000	50.0000
120.0100	2.1900	49.9700	50.0000
123.0100	2.6540	49.9600	50.0000
126.0100	3.7400	50.1300	50.0000
131.0200	4.2060	49.9100	50.0000
136.2100	5.1430	49.9500	50.0000
139.9110	6.8520	50.0000	50.0000
142.2020	7.9110	50.0200	50.0000
145.0100	17.4000	49.9300	50.0000
149.2100	24.3200	49.8800	50.0000
152.6100	30.5900	49.8700	50.0000
155.0100	37.4100	49.9600	50.0000
158.1200	43.0800	49.9900	50.0000
161.5100	53.5100	50.0300	50.0000
164.7100	67.2100	50.0300	50.0000
167.7100	83.0400	50.0700	50.0000
170.9000	99.4400	50.0700	50.0000
173.9060	121.5000	50.1100	50.0000
178.0000	170.0000	50.0100	50.0000
181.0200	224.5000	50.0000	50.0000
184.1000	289.8000	49.9800	50.0000
187.1000	373.1000	49.9900	50.0000
190.5000	505.7000	50.1500	50.0000
193.5100	640.8000	49.9300	50.0000
196.5100	865.3000	49.8800	50.0000

199.9110	1033.0000	49.9300	50.0000
203.0000	1385.0000	49.9900	50.0000
206.0160	1830.0000	50.0100	50.0000
209.2000	2466.0000	50.0200	50.0000
212.0000	3170.0000	50.0900	50.0000
214.5200	3897.0000	50.1100	50.0000
221.0050	6288.0000	50.0100	50.0000
224.1030	8313.0000	50.0300	50.0000
227.0100	11050.0000	50.1100	50.0000
230.1200	15300.0000	50.1400	50.0000
232.2100	17820.0000	50.1100	50.0000
232.4100	18240.0000	50.1100	50.0000
233.5100	20090.0000	50.0800	50.0000
235.0000	22970.0000	50.0700	50.0000
235.9100	24100.0000	50.0700	50.0000
236.1000	25110.0000	50.0700	50.0000
236.7100	25590.0000	50.0800	50.0000
236.9100	26480.0000	50.0800	50.0000
237.1100	27590.0000	50.0800	50.0000
237.4100	27820.0000	50.0900	50.0000
237.5000	27910.0000	50.0900	50.0000
237.6000	28280.0000	50.0900	50.0000
237.7000	29010.0000	50.0900	50.0000

Table C-11 Data For Run SDB6560

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
6.0140	0.0000	60.1600	60.0000
30.0100	0.0000	59.9500	60.0000
54.0100	0.0000	60.1700	60.0000
58.8100	0.0000	59.8800	60.0000
64.9100	0.0000	51.5000	50.0000
79.2070	1.3780	50.1200	50.0000
84.8100	2.0250	49.9600	50.0000
90.4100	3.0290	49.9100	50.0000
94.6100	4.8400	49.8100	50.0000
98.2000	5.9990	49.9200	50.0000
102.4000	8.5110	50.0800	50.0000
105.7200	10.3300	50.0500	50.0000
108.7100	13.3100	50.0300	50.0000
110.8100	15.3200	50.0200	50.0000
112.8100	18.1900	50.0300	50.0000
114.8000	21.7500	50.0400	50.0000
118.8000	30.5100	49.9700	50.0000
120.9110	115.0000	49.8100	50.0000
122.7070	158.9000	50.0400	50.0000
124.8080	175.7000	50.0100	50.0000
126.8010	207.4000	49.9500	50.0000
128.9100	247.0000	49.9700	50.0000
130.9100	313.4000	50.0100	50.0000
132.9100	374.5000	50.0200	50.0000
134.8100	471.1000	50.0900	50.0000
136.8000	557.7000	50.1000	50.0000
138.0000	639.9000	50.0900	50.0000
139.4100	745.7000	50.0900	50.0000
141.0100	901.2000	50.0700	50.0000
142.4100	1029.0000	50.0900	50.0000
144.0000	1259.0000	50.1200	50.0000
145.4000	1413.0000	50.1500	50.0000
147.1100	1828.0000	50.1600	50.0000

148.5100	2175.0000	50.1100	50.0000
150.4010	2341.0000	50.0800	50.0000
152.3120	3040.0000	50.1400	50.0000
154.9060	3936.0000	50.0100	50.0000
156.5050	4453.0000	50.0300	50.0000
157.9050	5349.0000	50.0300	50.0000
159.2100	6129.0000	50.0600	50.0000
160.8100	7704.0000	50.1000	50.0000
163.3000	9475.0000	50.2200	50.0000
164.8100	11740.0000	50.2700	50.0000
166.3100	13570.0000	50.3200	50.0000
167.2100	15280.0000	50.2500	50.0000
168.1000	16190.0000	50.2200	50.0000
169.0000	17840.0000	50.2000	50.0000
169.6100	18900.0000	50.2000	50.0000
170.2000	19900.0000	50.2000	50.0000
170.8100	20860.0000	50.1900	50.0000
171.2100	22760.0000	50.1800	50.0000
171.8000	23480.0000	50.1700	50.0000
172.1100	24080.0000	50.1500	50.0000
172.5100	25720.0000	50.1600	50.0000
173.0000	26810.0000	50.1400	50.0000
173.4200	27790.0000	50.1100	50.0000
173.7100	28510.0000	50.1100	50.0000
173.9100	29280.0000	50.1000	50.0000
174.0000	30820.0000	50.1000	50.0000
174.3100	31870.0000	50.1100	50.0000
174.5100	32180.0000	50.1200	50.0000
175.0100	34010.0000	50.1300	50.0000
175.6100	35330.0000	50.1200	50.0000
176.2100	35470.0000	50.1530	50.0000
177.0200	37480.0000	50.1500	50.0000
177.3100	38180.0000	50.1700	50.0000
177.4100	39060.0000	50.1600	50.0000
177.5100	40390.0000	50.1600	50.0000
177.6100	40777.0000	50.1500	50.0000

Table C-12 Data For Run RSDB6560

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
7.2010	0.0000	59.7000	60.0000
30.0100	0.0000	60.1200	60.0000
58.8100	0.0000	59.9000	60.0000
68.6427	0.6146	51.7800	50.0000
71.8560	0.8223	50.9500	50.0000
74.4100	1.0050	49.9500	50.0000
78.8900	1.4190	50.2000	50.0000
82.0900	1.7390	50.0200	50.0000
85.2900	2.2260	50.0200	50.0000
88.4800	2.8280	50.1100	50.0000
91.0500	3.4710	50.1000	50.0000
94.8900	4.5970	50.0600	50.0000
98.0900	5.8890	49.9100	50.0000
101.5200	8.6410	49.9700	50.0000
104.5200	12.1700	49.9600	50.0000
107.5100	14.3200	50.0500	50.0000
110.5100	18.0400	50.0900	50.0000
113.5100	23.2600	50.4200	50.0000
116.6100	30.1700	50.2500	50.0000
119.6100	39.1100	50.3600	50.0000

122.0000	48.6900	50.2800	50.0000
124.0100	58.5800	50.2600	50.0000
126.0050	145.3000	50.2900	50.0000
128.0160	171.1000	50.1300	50.0000
130.0000	230.1000	50.0000	50.0000
132.0100	296.3000	49.8800	50.0000
134.0040	353.7000	49.8500	50.0000
136.0100	448.1000	49.8400	50.0000
138.0200	555.2000	49.8100	50.0000
140.0000	704.9000	49.8800	50.0000
142.0100	862.0000	49.8800	50.0000
144.0000	1152.0000	49.9100	50.0000
146.0100	1421.0000	49.9800	50.0000
148.0200	1815.0000	49.9600	50.0000
150.0000	2260.0000	49.9600	50.0000
152.0100	2837.0000	50.0100	50.0000
154.0050	3612.0000	49.9900	50.0000
156.0150	4543.0000	50.0600	50.0000
158.0000	5646.0000	49.9800	50.0000
159.0050	6288.0000	50.0400	50.0000
160.0100	6786.0000	49.9800	50.0000
161.0160	7441.0000	49.9700	50.0000
162.0040	8443.0000	49.9900	50.0000
163.0000	8938.0000	49.9800	50.0000
164.0100	9819.0000	50.0200	50.0000
165.0100	11090.0000	50.0600	50.0000
166.0200	12070.0000	50.0300	50.0000
167.0000	13070.0000	50.0600	50.0000
168.0000	14820.0000	50.1300	50.0000
169.0100	16120.0000	50.1800	50.0000
170.0100	17330.0000	50.1400	50.0000
171.0200	18820.0000	50.0800	50.0000
172.0000	21080.0000	50.1000	50.0000
173.0000	23010.0000	50.1000	50.0000
174.0100	24670.0000	50.0900	50.0000
174.5200	24950.0000	50.0800	50.0000
174.7100	25650.0000	50.0900	50.0000
174.9100	26580.0000	50.0800	50.0000
175.0100	26580.0000	50.0800	50.0000
175.1100	28550.0000	50.0700	50.0000
175.2100	29350.0000	50.0700	50.0000

Table C-13 Data For Run SIC56100

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.4060	0.0000	49.8600	50.0000
25.2100	0.0000	49.9600	50.0000
50.4100	0.0000	50.0400	50.0000
75.6000	0.0000	49.9500	50.0000
99.6100	0.3715	50.1100	50.0000
102.0000	0.6464	50.9500	60.0000
104.4000	0.8657	54.0600	60.0000
106.2030	1.8830	56.7400	60.0000
110.1080	2.9820	60.1700	60.0000
112.2010	4.2000	60.2700	60.0000
114.0130	5.9900	60.3000	60.0000
116.1100	9.2160	60.1200	60.0000
118.2200	14.9300	59.8900	60.0000
120.0000	26.9300	59.8100	60.0000
121.5490	33.7700	59.8500	60.0000

123.0810	45.2300	59.9200	60.0000
123.5100	49.3900	59.9300	60.0000
124.0700	55.7200	59.9500	60.0000
124.4820	61.5000	59.9600	60.0000
124.9100	68.3300	59.9800	60.0000
125.6154	135.1000	59.9800	60.0000
126.0108	134.9000	60.0100	60.0000
126.5050	150.6000	60.0300	60.0000
127.0160	180.5000	60.0500	60.0000
127.2140	188.0000	60.0400	60.0000
127.4110	204.6000	60.0500	60.0000
127.6090	209.2000	60.0600	60.0000
127.8070	230.4000	60.0600	60.0000
128.0040	238.6000	60.0500	60.0000
128.2020	246.2000	60.0600	60.0000
128.4000	275.2000	60.0700	60.0000
128.6140	305.4000	60.0600	60.0000
128.8120	327.4000	60.0600	60.0000
129.0100	340.9000	60.0700	60.0000
129.2070	369.6000	60.0800	60.0000
129.4050	395.8000	60.0900	60.0000
129.6030	431.1000	60.1000	60.0000
129.8010	455.6000	60.1000	60.0000
130.0150	531.5000	60.1100	60.0000
130.2130	560.9000	60.1300	60.0000
130.4100	646.2000	60.1400	60.0000
130.6150	669.3000	60.1400	60.0000
130.8130	728.1000	60.1300	60.0000
131.0110	786.7000	60.1300	60.0000
131.2090	897.4000	60.1400	60.0000
131.4060	983.3000	60.1500	60.0000
131.6040	1084.0000	60.1600	60.0000
131.8020	1202.0000	60.1600	60.0000
132.0160	1422.0000	60.1800	60.0000
132.2140	1484.0000	60.1900	60.0000
132.4120	1673.0000	60.2000	60.0000
132.6090	1770.0000	60.2300	60.0000
132.8070	1907.0000	60.2500	60.0000
133.0050	2117.0000	60.2600	60.0000
133.2020	2396.0000	60.3000	60.0000
133.4000	3469.0000	60.3800	60.0000
133.6140	7273.0000	60.4100	60.0000
133.8120	10840.0000	60.3900	60.0000
134.0100	15230.0000	60.3300	60.0000
134.3070	26400.0000	60.2500	60.0000

Table C-14 Data For Run SIC56150

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.2030	0.0000	49.9100	50.0000
31.0100	0.0000	49.9900	50.0000
61.0000	0.0000	50.0800	50.0000
92.0560	0.1926	50.0700	50.0000
120.5200	1.2080	49.9300	50.0000
149.8100	10.8700	49.9300	50.0000
152.5100	12.3200	54.0100	60.0000
154.6100	13.1800	57.1300	60.0000
156.7200	15.8500	59.6700	60.0000
158.8100	20.1100	60.2400	60.0000
160.9000	25.9500	60.1300	60.0000

162.5098	57.7900	60.0800	60.0000
164.0090	63.6200	60.0300	60.0000
166.0030	94.7300	60.0700	60.0000
168.0040	123.0000	60.0200	60.0000
170.0140	178.8000	59.9500	60.0000
172.0200	251.5000	59.9700	60.0000
174.0100	355.6000	60.0300	60.0000
176.0000	532.2000	60.0600	60.0000
178.0000	795.1000	60.1500	60.0000
179.5000	1073.0000	60.1800	60.0000
180.9100	1616.0000	60.2300	60.0000
182.6030	2123.0000	60.2300	60.0000
183.5090	2748.0000	60.1800	60.0000
185.0090	4320.0000	60.2000	60.0000
185.5030	5150.0000	60.2200	60.0000
186.0140	5962.0000	60.2700	60.0000
186.2120	6599.0000	60.2900	60.0000
186.6140	7219.0000	60.3200	60.0000
186.8120	8070.0000	60.3300	60.0000
187.0100	8526.0000	60.3100	60.0000
187.2070	8807.0000	60.2400	60.0000
187.3060	9371.0000	60.2200	60.0000
187.5040	9903.0000	60.1200	60.0000
187.7020	10450.0000	60.0200	60.0000
187.9160	11700.0000	59.9600	60.0000
188.1140	12780.0000	59.9200	60.0000
188.2130	13020.0000	59.9000	60.0000
188.3110	13450.0000	59.9000	60.0000
188.5090	13610.0000	59.8900	60.0000
188.6080	13810.0000	59.9000	60.0000
188.8060	13920.0000	59.9300	60.0000
188.9040	14180.0000	59.9500	60.0000
189.0030	14610.0000	59.3970	60.0000
189.1020	15520.0000	59.9800	60.0000
189.2010	15660.0000	60.0000	60.0000
189.3000	16970.0000	60.0200	60.0000
189.4150	19360.0000	60.0300	60.0000
189.6130	19560.0000	60.0500	60.0000
189.7120	19740.0000	60.0700	60.0000
189.8110	21100.0000	60.0700	60.0000
189.9100	23020.0000	60.0700	60.0000
190.0080	25580.0000	60.0800	60.0000
190.1070	27770.0000	60.0900	60.0000

Table C-15 Data For Run SIC6740

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.7020	0.0000	60.1600	60.0000
18.0100	0.0000	60.0300	60.0000
38.0930	0.1431	59.8000	60.0000
42.0800	0.4786	61.2100	70.0000
45.4010	0.5001	69.8600	70.0000
48.0050	0.6395	70.0500	70.0000
51.2020	1.4560	70.3200	70.0000
54.0030	3.0220	70.1000	70.0000
56.0130	4.9870	69.7700	70.0000
57.3100	9.9280	70.0200	70.0000
58.4000	15.9100	70.1400	70.0000
59.5000	25.3500	70.1100	70.0000
60.0200	34.6600	70.1400	70.0000

60.6100	46.7100	70.1300	70.0000
61.0000	66.3100	70.1000	70.0000
61.5100	94.1400	70.0800	70.0000
62.0000	117.8000	70.1000	70.0000
62.5100	167.2000	70.1200	70.0000
63.0100	353.1000	70.1200	70.0000
63.2000	469.8000	70.1300	70.0000
63.4000	663.8000	70.1200	70.0000
63.6100	996.4000	60.1200	70.0000
63.8100	1492.0000	70.1100	70.0000
64.0100	1889.0000	70.1000	70.0000
64.2100	2303.0000	70.0900	70.0000
64.4100	2861.0000	70.1100	70.0000
64.6000	3307.0000	70.1300	70.0000
64.8000	4211.0000	70.1700	70.0000
65.0200	5678.0000	70.2000	70.0000
65.2100	7495.0000	70.2300	70.0000
65.4100	9871.0000	70.2400	70.0000
65.5100	10890.0000	70.2500	70.0000
65.6100	11960.0000	70.2800	70.0000
65.7100	13890.0000	70.3200	70.0000
65.8100	14230.0000	70.3300	70.0000
65.9100	16140.0000	70.3400	70.0000
66.0000	16210.0000	70.3500	70.0000
66.1000	17450.0000	70.3800	70.0000
66.2000	17820.0000	70.4100	70.0000
66.3000	19230.0000	70.4400	70.0000
66.5100	20460.0000	70.4800	70.0000
66.6100	21940.0000	70.4800	70.0000
66.7100	22070.0000	70.5000	70.0000
66.8100	22570.0000	70.5100	70.0000
66.9100	22950.0000	70.5200	70.0000
67.0000	23220.0000	70.5200	70.0000
67.0700	23654.0000	70.5000	70.0000
67.1300	24780.0000	70.4800	70.0000
67.1800	25190.0000	70.4800	70.0000
67.2500	25220.0000	70.4500	70.0000
67.3100	25524.0000	70.4300	70.0000
67.3700	26140.0000	70.4200	70.0000
67.4300	26420.0000	70.4000	70.0000
67.4800	28010.0000	70.3800	70.0000

Table C-16 Data For Run SIC6760

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
3.5100	0.0000	59.8500	60.0000
23.1000	0.0000	59.9700	60.0000
40.4000	0.0000	60.0500	60.0000
48.3625	0.3828	59.7900	60.0000
49.4500	0.4258	59.8600	60.0000
51.2510	0.5234	59.8200	60.0000
53.0470	0.6755	59.8800	60.0000
54.4810	0.8285	59.9800	60.0000
55.5690	1.0360	59.9800	60.0000
57.7270	1.3960	59.9000	60.0000
59.8900	2.0240	60.0700	60.0000
62.0400	3.0820	61.2300	70.0000
64.2000	4.2050	64.4200	70.0000
66.0000	5.6900	67.5830	70.0000
66.5100	6.7910	68.0200	70.0000

67.0100	7.8100	68.8600	70.0000
67.5200	8.8670	69.4500	70.0000
68.0100	9.9060	70.0100	70.0000
68.5000	11.2900	70.1200	70.0000
69.0200	12.9000	70.1400	70.0000
69.5100	14.6200	70.2000	70.0000
70.0000	17.0900	70.2000	70.0000
70.5100	19.9400	70.2300	70.0000
71.0000	22.9700	70.2300	70.0000
71.5300	27.9900	70.2200	70.0000
72.0100	33.5700	70.1800	70.0000
72.5300	41.9100	70.1500	70.0000
73.0000	52.3200	70.1300	70.0000
73.5100	105.8000	70.1000	70.0000
74.0000	177.9000	70.1500	70.0000
74.5200	297.0000	70.2100	70.0000
75.0100	416.1000	70.2300	70.0000
75.5000	479.4000	70.2400	70.0000
76.0200	581.9000	70.2800	70.0000
76.5100	1429.0000	70.3500	70.0000
76.7100	1841.0000	70.4000	70.0000
76.9100	2620.0000	70.4700	70.0000
77.1000	3728.0000	70.4800	70.0000
77.2000	4646.0000	70.5000	70.0000
77.3000	5630.0000	70.4900	70.0000
77.4200	7446.0000	70.5200	70.0000
77.5100	9459.0000	70.5200	70.0000
77.6100	12330.0000	70.5100	70.0000
77.7100	14350.0000	70.4800	70.0000
77.8100	16120.0000	70.4400	70.0000
77.9100	17850.0000	70.4000	70.0000
78.0000	19240.0000	70.3600	70.0000
78.1000	20500.0000	70.3400	70.0000
78.2000	21710.0000	70.3100	70.0000
78.3000	22210.0000	70.2900	70.0000
78.4200	23550.0000	70.2100	70.0000
78.5100	24680.0000	70.2100	70.0000
78.6100	25210.0000	70.1800	70.0000
78.7100	26340.0000	70.1000	70.0000
78.8100	27720.0000	70.0600	70.0000

Table C-17 Data For Run RSIC6760

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
7.0030	0.0000	60.0300	60.0000
15.0000	0.0000	59.9800	60.0000
25.0000	0.0000	60.0200	60.0000
35.0000	0.0000	59.9800	60.0000
39.0630	0.1407	60.0000	60.0000
41.9470	0.4876	61.4200	70.0000
44.0230	0.4420	63.0000	70.0000
46.0090	0.7876	67.3100	70.0000
49.0050	0.6395	70.1200	70.0000
50.0100	0.7618	70.1400	70.0000
51.0150	0.9762	70.1500	70.0000
52.0040	1.5150	70.1400	70.0000
53.0090	2.1160	70.1200	70.0000
54.0140	2.4490	70.1300	70.0000
55.0030	3.0220	70.1200	70.0000
56.0080	3.5920	70.1300	70.0000

57.0130	4.9870	70.1200	70.0000
58.0000	6.3570	70.1500	70.0000
59.0100	11.2700	70.1000	70.0000
60.0100	21.7100	70.0800	70.0000
60.5000	25.3500	70.0600	70.0000
61.0200	34.6600	70.0400	70.0000
61.5100	41.1500	70.0300	70.0000
62.0000	66.3100	70.0300	70.0000
62.5100	94.1400	70.1000	70.0000
63.0000	117.8000	70.0000	70.0000
63.5100	167.2000	69.9800	70.0000
64.0100	353.1000	69.9700	70.0000
64.5200	809.8000	69.9600	70.0000
65.0100	1889.0000	69.9500	70.0000
65.5000	3166.0000	69.9200	70.0000
66.0200	5678.0000	69.9000	70.0000
66.4100	9871.0000	69.8600	70.0000
66.8100	14230.0000	69.8400	70.0000
67.1000	17450.0000	69.8000	70.0000
67.3000	19230.0000	69.8100	70.0000
67.5100	20460.0000	69.8200	70.0000
67.7100	22070.0000	69.8300	70.0000
67.9100	22950.0000	69.8400	70.0000
68.0700	23650.0000	69.8500	70.0000
68.2500	25220.0000	69.8700	70.0000
68.3100	25520.0000	69.8800	70.0000
68.3700	26140.0000	69.9000	70.0000

Table C-18 Data For Run SDC7615

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.7020	0.0000	69.8500	70.0000
9.0130	0.0000	70.1900	70.0000
13.5100	0.0000	70.1700	70.0000
18.0100	0.0000	63.1000	60.0000
27.0100	0.0000	60.0300	60.0000
41.5000	0.5922	59.8400	60.0000
43.0160	0.6074	59.8500	60.0000
44.5150	0.7972	59.8100	60.0000
46.0150	0.9672	59.8200	60.0000
47.5050	2.2260	59.8400	60.0000
49.9110	2.6580	59.8300	60.0000
51.7100	3.1490	59.8900	60.0000
53.7000	4.1630	60.1400	60.0000
55.5100	5.2660	60.1500	60.0000
56.3000	6.1200	60.1300	60.0000
57.0142	7.9330	60.2100	60.0000
57.5085	8.6440	60.1900	60.0000
58.2010	10.0100	60.2100	60.0000
58.7120	11.1800	60.1900	60.0000
59.2060	12.7200	60.1900	60.0000
59.7000	13.8800	60.1800	60.0000
60.3150	16.3900	60.1700	60.0000
60.8100	18.3400	60.1800	60.0000
61.2050	20.2700	60.2000	60.0000
62.0140	33.9500	60.1800	60.0000
62.5080	59.7400	60.1800	60.0000
63.0030	73.0700	60.1500	60.0000
63.5140	74.0100	60.1000	60.0000
64.0080	93.5900	59.9800	60.0000

64.5020	103.4000	59.9000	60.0000
65.0130	114.6000	59.8500	60.0000
65.5070	134.2000	59.8400	60.0000
66.0020	146.8000	59.7900	60.0000
66.5120	181.8000	59.8100	60.0000
67.0100	201.2000	59.9000	60.0000
67.5100	231.7000	59.9500	60.0000
68.0000	274.1000	60.1000	60.0000
68.5100	338.2000	60.1200	60.0000
69.0100	368.8000	60.1400	60.0000
69.5000	437.2000	60.1100	60.0000
70.0100	516.0000	60.1400	60.0000
70.5100	611.8000	60.1400	60.0000
71.0000	771.3000	60.1100	60.0000
71.5100	950.1000	60.1000	60.0000
72.0100	1188.0000	60.1200	60.0000
72.5100	1503.0000	60.1300	60.0000
73.0000	1823.0000	60.1500	60.0000
73.5100	2207.0000	60.1400	60.0000
74.0100	2701.0000	60.1500	60.0000
74.5000	3620.0000	60.1700	60.0000
75.0100	5201.0000	60.2000	60.0000
75.5100	5862.0000	60.2500	60.0000
76.0000	6894.0000	60.5500	60.0000
76.5100	8451.0000	60.3100	60.0000
77.0100	9634.0000	60.1000	60.0000
77.5100	13810.0000	59.8500	60.0000
78.0000	17610.0000	59.7500	60.0000
78.2000	19650.0000	59.7900	60.0000
78.4100	22550.0000	59.9000	60.0000
78.5100	24580.0000	59.9100	60.0000
78.6100	26790.0000	59.9500	60.0000
78.7100	28580.0000	60.0000	60.0000
78.8100	30630.0000	60.0500	60.0000

Table C-19 Data For Run SDC7625

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.1090	0.0000	70.3200	70.0000
10.5100	0.0000	70.0900	70.0000
14.0100	0.0000	70.1300	70.0000
21.0100	0.0000	70.2200	70.0000
35.1500	0.3965	59.9900	60.0000
35.7100	0.6012	59.9400	60.0000
36.2700	0.9036	59.9100	60.0000
37.4283	1.8570	59.8000	60.0000
38.2690	2.1470	59.8100	60.0000
39.1090	2.6060	59.9000	60.0000
40.0810	3.0910	60.0000	60.0000
40.5100	3.4770	60.2300	60.0000
41.0700	3.9080	60.2100	60.0000
41.4820	4.1160	60.2300	60.0000
42.0420	4.8020	60.2400	60.0000
42.4710	5.2820	60.2600	60.0000
43.0310	6.0340	60.2400	60.0000
43.5910	7.0430	60.1700	60.0000
44.1150	9.4460	60.0700	60.0000
44.6100	18.8500	59.9900	60.0000
45.0050	25.8900	59.9500	60.0000
45.3020	27.3200	59.9000	60.0000

46.7020	33.1400	59.8500	60.0000
47.2100	38.1000	59.8800	60.0000
47.6100	41.6400	59.9600	60.0000
48.0000	48.4100	60.0200	60.0000
48.4200	55.2000	60.0600	60.0000
49.0000	62.7400	60.1100	60.0000
49.5100	78.0900	60.2400	60.0000
50.0100	95.6900	60.2300	60.0000
50.5200	113.8000	60.1900	60.0000
51.0100	132.7000	60.1900	60.0000
51.5000	164.7000	60.0500	60.0000
52.0200	211.4000	59.9600	60.0000
52.5100	298.2000	59.9000	60.0000
53.0000	348.9000	59.9600	60.0000
53.5100	448.5000	60.1500	60.0000
53.9100	510.5000	60.1400	60.0000
54.7143	832.5000	60.2000	60.0000
55.0108	930.5000	60.1500	60.0000
55.5050	1492.0000	59.9500	60.0000
56.0160	2116.0000	60.0000	60.0000
56.5100	2404.0000	60.1500	60.0000
57.0050	3229.0000	60.2500	60.0000
57.4000	4044.0000	60.3800	60.0000
57.6140	5128.0000	60.4500	60.0000
57.8120	5808.0000	60.5100	60.0000
58.0100	6470.0000	60.5200	60.0000
58.4050	7175.0000	60.5200	60.0000
58.8010	8959.0000	60.3500	60.0000
58.9160	9875.0000	60.2900	60.0000
59.0150	10700.0000	60.2500	60.0000
59.1140	12360.0000	60.1900	60.0000
59.2130	14480.0000	60.1300	60.0000
59.3110	16100.0000	60.1000	60.0000
59.4100	17510.0000	60.0500	60.0000
59.5000	18870.0000	60.0100	60.0000
59.6150	21460.0000	60.0000	60.0000
59.7140	24420.0000	59.9500	60.0000
59.8130	26980.0000	59.9300	60.0000

Table C-20 Data For Run SDC6530

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
2.4060	0.0000	60.2500	60.0000
10.8100	0.0000	59.9400	60.0000
20.4200	0.0000	59.8400	60.0000
29.0140	0.0000	59.9400	60.0000
35.0100	0.0000	51.6900	50.0000
53.0100	0.0000	49.9100	50.0000
65.0000	0.0329	49.8800	50.0000
70.0550	0.5218	49.9400	50.0000
75.5090	0.8223	49.9900	50.0000
79.9200	1.0960	50.0200	50.0000
86.0900	1.7850	50.0000	50.0000
91.5200	2.8380	50.0400	50.0000
95.6100	4.1610	50.0700	50.0000
101.0500	7.0750	50.0600	50.0000
103.0900	8.6850	50.0900	50.0000
106.4900	12.4300	50.0500	50.0000
109.2000	16.5000	50.0700	50.0000
112.6200	23.7000	50.1100	50.0000

115.3300	31.4200	50.0400	50.0000
118.0100	51.5200	50.0300	50.0000
120.0000	66.5200	50.0800	50.0000
122.0100	85.7200	50.0700	50.0000
124.0100	110.9000	50.0800	50.0000
126.0000	140.6000	50.1800	50.0000
128.0100	175.4000	50.2300	50.0000
130.0073	394.0000	50.3100	50.0000
130.9140	469.9000	50.3600	50.0000
132.0010	548.0000	50.3200	50.0000
133.0060	687.9000	50.2900	50.0000
133.9130	811.2000	50.3000	50.0000
135.0070	924.7000	50.2800	50.0000
136.0130	1078.0000	50.2900	50.0000
137.0010	1370.0000	50.2600	50.0000
138.0060	1603.0000	50.3000	50.0000
139.0110	1883.0000	50.3800	50.0000
140.0100	2195.0000	50.1500	50.0000
141.0100	2661.0000	50.0900	50.0000
142.0000	3153.0000	50.0500	50.0000
142.1000	3277.0000	50.0500	50.0000
143.0100	3736.0000	50.0500	50.0000
144.0100	4431.0000	50.0900	50.0000
145.1100	5438.0000	50.1100	50.0000
146.0100	6374.0000	50.1100	50.0000
147.0000	7844.0000	50.1300	50.0000
148.0100	9321.0000	50.1300	50.0000
149.0100	11160.0000	50.1700	50.0000
150.0100	13510.0000	50.2100	50.0000
151.0100	15840.0000	50.2900	50.0000
151.4100	17630.0000	50.3500	50.0000
151.9000	18710.0000	50.4000	50.0000
152.5100	21610.0000	50.2500	50.0000
152.7100	22200.0000	50.2000	50.0000
153.0100	22930.0000	50.0000	50.0000
153.1100	23480.0000	50.1500	50.0000
153.2000	24900.0000	50.1000	50.0000
153.4000	25720.0000	50.0900	50.0000
153.6200	26770.0000	50.0500	50.0000
153.8100	28060.0000	50.0300	50.0000

Table C-21 Data For Run SDC6560

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
6.0140	0.0000	59.9300	60.0000
15.6000	0.0000	60.1700	60.0000
30.0100	0.0000	59.9800	60.0000
45.4220	0.1720	60.0000	60.0000
65.0300	1.6370	52.1300	50.0000
69.5000	3.0790	50.0000	50.0000
72.5200	7.3190	50.1300	50.0000
74.9000	10.9300	50.1900	50.0000
77.0100	14.9800	50.1800	50.0000
80.0100	25.4900	50.0900	50.0000
83.0100	42.0400	50.0100	50.0000
84.5000	55.0500	50.0400	50.0000
86.0200	143.0000	50.0300	50.0000
87.0000	177.0000	50.1200	50.0000
88.0100	219.5000	50.1300	50.0000

89.0100	261.8000	50.1900	50.0000
90.0100	323.4000	50.2300	50.0000
91.0200	395.8000	50.2300	50.0000
92.0000	494.1000	50.1900	50.0000
93.0100	611.7000	50.0800	50.0000
94.0100	776.6000	50.0000	50.0000
95.5130	953.6000	49.9000	50.0000
96.0073	1060.0000	49.9500	50.0000
97.0130	1384.0000	50.0800	50.0000
98.0010	1665.0000	50.2000	50.0000
99.0060	2139.0000	50.1800	50.0000
100.0110	2651.0000	50.1500	50.0000
100.5130	2880.0000	50.1500	50.0000
101.0080	3179.0000	50.1500	50.0000
101.5020	3502.0000	50.1700	50.0000
102.0130	4083.0000	50.1900	50.0000
102.5070	4619.0000	50.2100	50.0000
103.0010	5013.0000	50.1900	50.0000
103.5120	5745.0000	50.1700	50.0000
104.0060	6399.0000	50.1500	50.0000
104.5010	7284.0000	50.1400	50.0000
105.0110	8089.0000	50.1400	50.0000
105.5100	8909.0000	50.1500	50.0000
106.0100	9863.0000	50.1400	50.0000
106.5000	10950.0000	50.1500	50.0000
107.0100	11920.0000	50.1500	50.0000
107.5100	13190.0000	50.2000	50.0000
108.0000	15100.0000	50.2700	50.0000
108.5100	17000.0000	50.2900	50.0000
109.0100	19030.0000	50.3400	50.0000
109.4000	20360.0000	50.3200	50.0000
109.8100	22930.0000	50.2800	50.0000
110.0100	23200.0000	50.2500	50.0000
110.2000	24290.0000	50.2400	50.0000
110.3200	24580.0000	50.2300	50.0000
110.4100	25870.0000	50.2300	50.0000
110.5100	25980.0000	50.2300	50.0000
110.6100	27000.0000	50.2200	50.0000
110.7100	27110.0000	50.2200	50.0000

Table C-22 Data For Run RSDC7615

Time (min)	Viscosity (Pa.s)	Temperature (°C)	Set Point (°C)
3.7130	0.0000	69.9300	70.0000
8.5080	0.0000	69.8800	70.0000
14.9000	0.0000	70.0600	70.0000
18.9100	0.0000	60.7700	60.0000
24.5100	0.0000	59.7400	60.0000
30.9000	0.0000	59.9000	60.0000
40.5132	0.4987	59.9500	60.0000
42.0130	0.6091	59.9000	60.0000
42.9020	0.7130	59.9000	60.0000
44.1050	0.8425	59.9500	60.0000
45.0120	0.9585	59.9900	60.0000
45.9030	1.0810	60.0300	60.0000
47.1060	1.3200	60.0400	60.0000
48.0120	1.4980	60.0300	60.0000
48.9020	1.7610	59.9900	60.0000
50.1050	2.1170	59.9600	60.0000
51.0100	2.4860	60.0300	60.0000

51.9000	2.9200	59.9200	60.0000
53.1000	3.6830	59.9200	60.0000
54.0100	4.3490	59.9400	60.0000
54.9000	5.1820	59.9400	60.0000
56.0100	7.1850	60.0300	60.0000
57.0100	8.6700	60.0700	60.0000
58.0000	10.8200	60.1000	60.0000
59.0100	13.4200	60.1000	60.0000
60.0100	16.8300	60.1300	60.0000
61.0000	21.2900	60.1000	60.0000
62.0100	27.3600	59.9900	60.0000
63.0100	35.7200	59.9200	60.0000
64.0200	47.4400	59.9900	60.0000
65.0000	63.8700	60.1200	60.0000
66.0100	247.4000	60.2300	60.0000
66.9100	321.1000	60.4100	60.0000
68.0000	486.2000	60.3100	60.0000
69.5943	765.3000	60.2900	60.0000
70.0722	980.7000	60.0500	60.0000
70.5500	1147.0000	60.0800	60.0000
71.0280	1407.0000	60.0000	60.0000
71.5880	1745.0000	60.1000	60.0000
71.9840	2204.0000	60.1200	60.0000
72.5440	2792.0000	60.1600	60.0000
73.0220	3578.0000	60.1700	60.0000
73.5120	4281.0000	60.1700	60.0000
73.9900	5623.0000	60.1900	60.0000
74.5500	7577.0000	60.2100	60.0000
75.1100	11110.0000	60.2400	60.0000
75.3410	12100.0000	60.2500	60.0000
75.5880	12980.0000	60.2800	60.0000
75.7530	14470.0000	60.2600	60.0000
75.9840	16170.0000	60.2400	60.0000
76.1480	18450.0000	60.2100	60.0000
76.3130	20480.0000	60.1700	60.0000
76.4620	23580.0000	60.1500	60.0000
76.5440	24870.0000	60.1500	60.0000
76.6260	25900.0000	60.1300	60.0000
76.7090	29410.0000	60.1000	60.0000
76.7910	30300.0000	60.0900	60.0000
76.8740	31820.0000	60.0800	60.0000
76.9560	37670.0000	60.0500	60.0000